

# **Studies on Flux Decline Phenomena in Ultrafiltration of Sugarcane Juice for Recovery of Sugar**

*A Thesis submitted  
In Partial fulfilment of the Requirements  
for the degree of  
Master of Technology*

*by  
Shilpi Agarwal*

*to the*

**Department of Chemical Engineering  
Indian Institute of Technology, Kanpur  
January 1996**

13 MAY 1996

CENTRAL LIBRARY  
IIT KANPUR

Call No. A. 121482



A121482

## CERTIFICATE

This is to certify that the work entitled, 'STUDIES ON LIMITING FLUX PHENOMENA IN ULTRAFILTRATION OF SUGAR CANE JUICE FOR RECOVERY OF SUGAR' has been carried out under our supervision by SHILPI AGARWAL and that it has not been submitted elsewhere for a Degree.

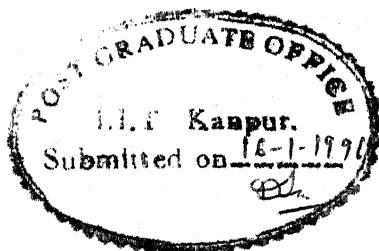
January, 1996.

J. Gehlawat

Dr J. K. Gehlawat,  
Professor,  
Department of Chemical Engineering  
Indian Institute of Technology.  
Kanpur - 208016. India.

P.K. Bhattacharya

Dr P. K. Bhattacharya, 16/1  
Professor,  
Department of Chemical Engineering  
Indian Institute of Technology  
Kanpur - 208016. India.



## Acknowledgement

Looking at my thesis I find it hard to believe that the thesis has finally taken shape in black and white. This would have been impossible without the effort of few people who helped me directly or indirectly.

First of all I wish to express my profound sense of gratitude to my thesis advisors Dr J. K. Gehlawat and Dr P. K. Bhattacharya for enlightening me in the field of research. Its my pleasure to acknowledge Dr J. K. G., for introducing me to the subject and helping me in the time of need. Its difficult to me to express my gratitude to Dr P. K. B. in words. He was to me a friend, philosopher and guide. It is him who made my work go uninterrupted despite of many obstacles which at many times made me doubt about the completion of the work.

I'm thankful to my labmates Mr Subir Bhattacharya, Mr Shishendu and Manoranjan Shymal for helping me deal with the difficulties of the experimental work. I'm grateful to Mr R. Chandra and Mr J. S. Virdi for their kind cooperation in handling the technical problems during experimental work. I'm highly indebted to all my friends Anasuya, Swati, Vineeta, Ipsita, Poonam and Ila for making my stay in IIT Kanpur, a memorable one. Here I wish to specially mention the name of Anasuya, without whom I wonder I would've been able to complete my thesis on time.

I'm thankful to Dr Ashutosh sharma for allowing me to use his PC and forgiving me for destroying the same. Lastly, I'm grateful to my parents, sisters and Madhukar for their constant encouragement and support.

Shilpi Agarwal

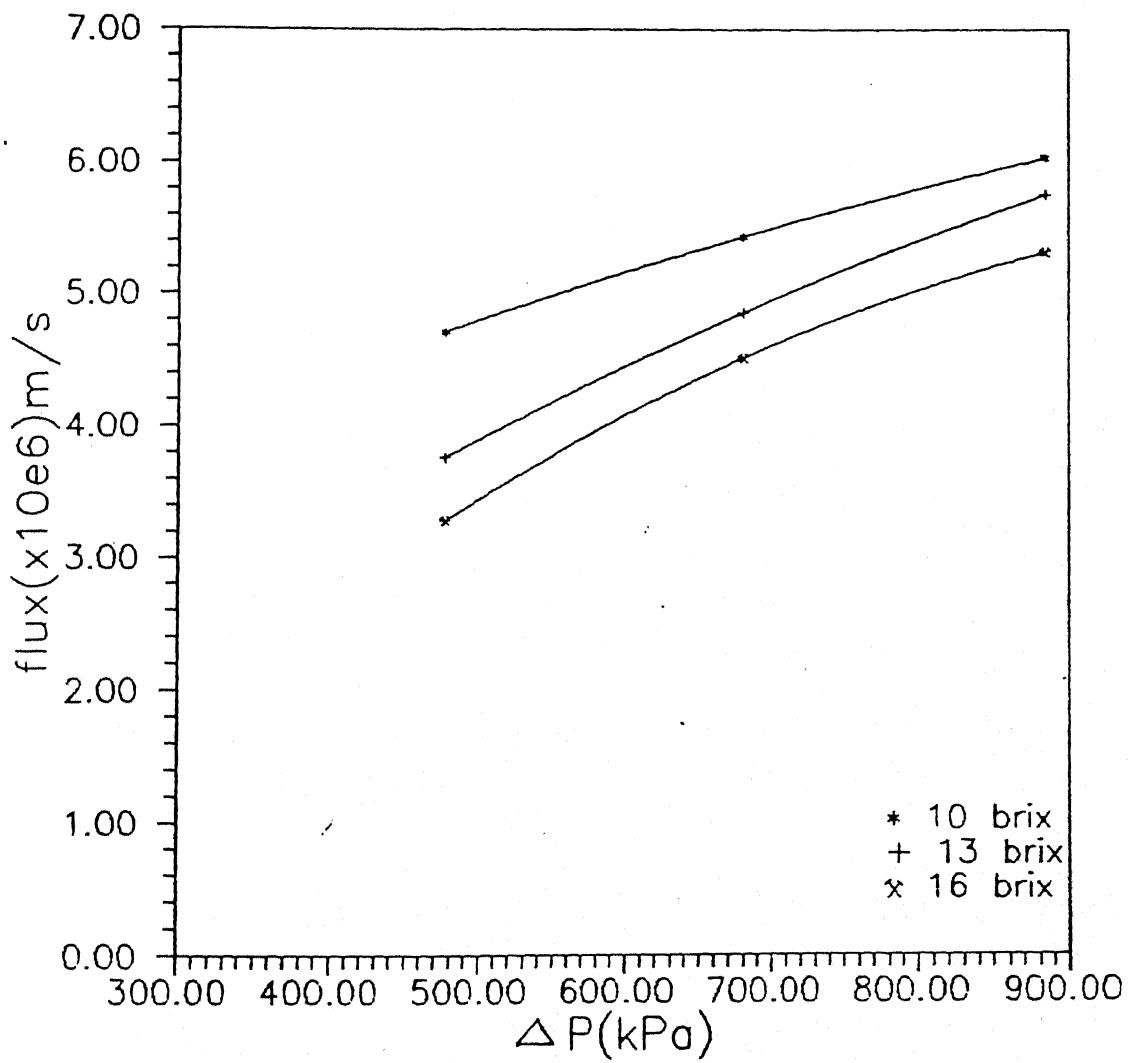


Fig 5.2 Effect of pressure on permeate flux for a 10000 membrane

## Contents

	Page no.
List of figures	vi
List of tables	viii
Nomenclature	iX
Abstract	Xii
<b>1. Introduction</b>	<b>1</b>
<b>2. Literature cited</b>	<b>6</b>
2.1 Industrial process for sugar manufacture	6
2.2 Review of membrane system tested	9
2.3 Review of studies on concentration polarisation UF	12
<b>3. Theoretical consideration</b>	<b>13</b>
3.1 Ultrafiltration	13
3.2 Flux	13
3.3 Concentration polarisation	14
3.4 Rejection	18
3.5 Molecular weight cut-off profile	19
3.6 Estimation of additional resistance offered by the membrane due to retention of solutes	19
3.7 Ultrafiltration of cane juice	22

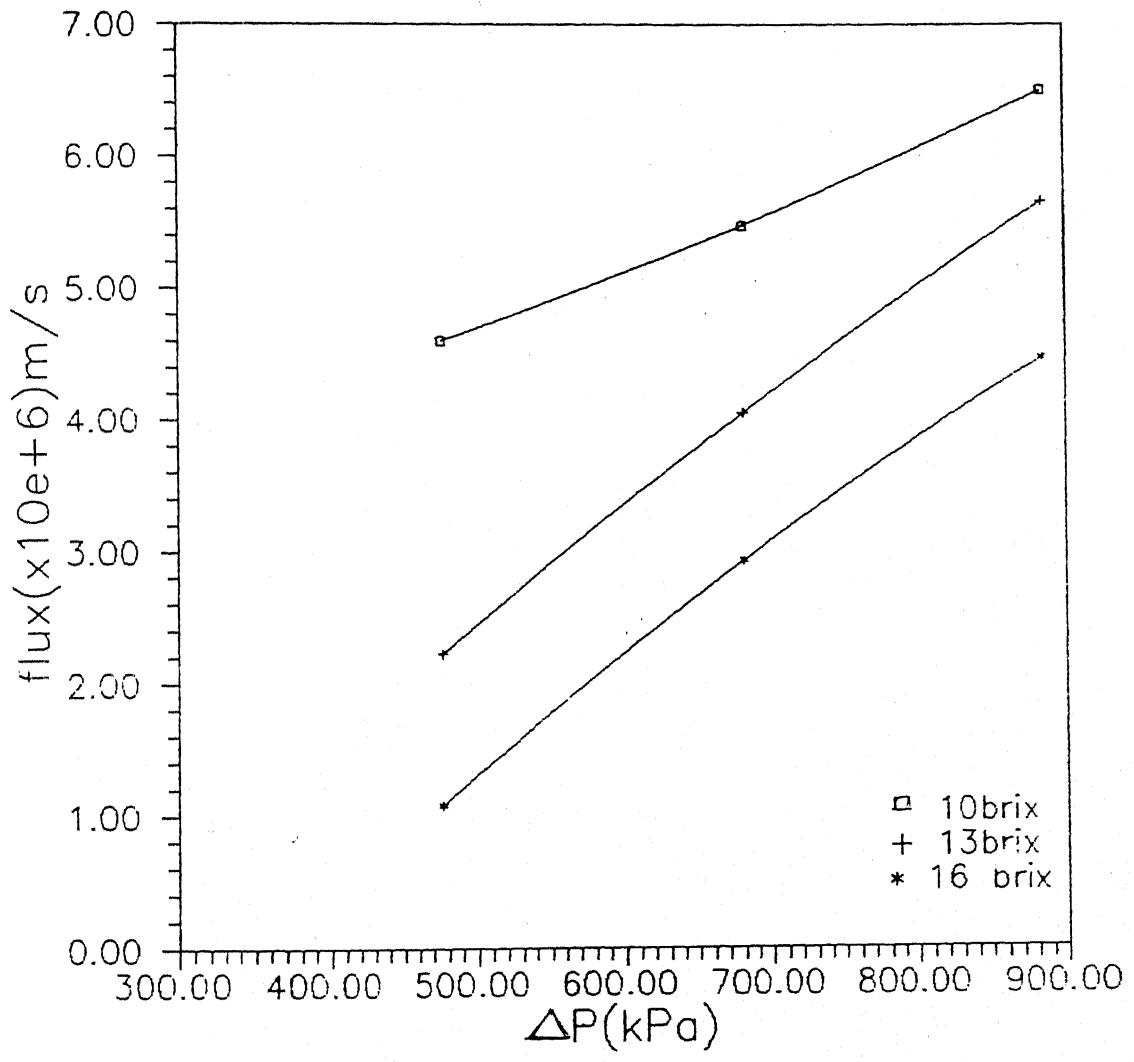


Fig 5.3 Effect of pressure on permeate flux for a 15000 membrane

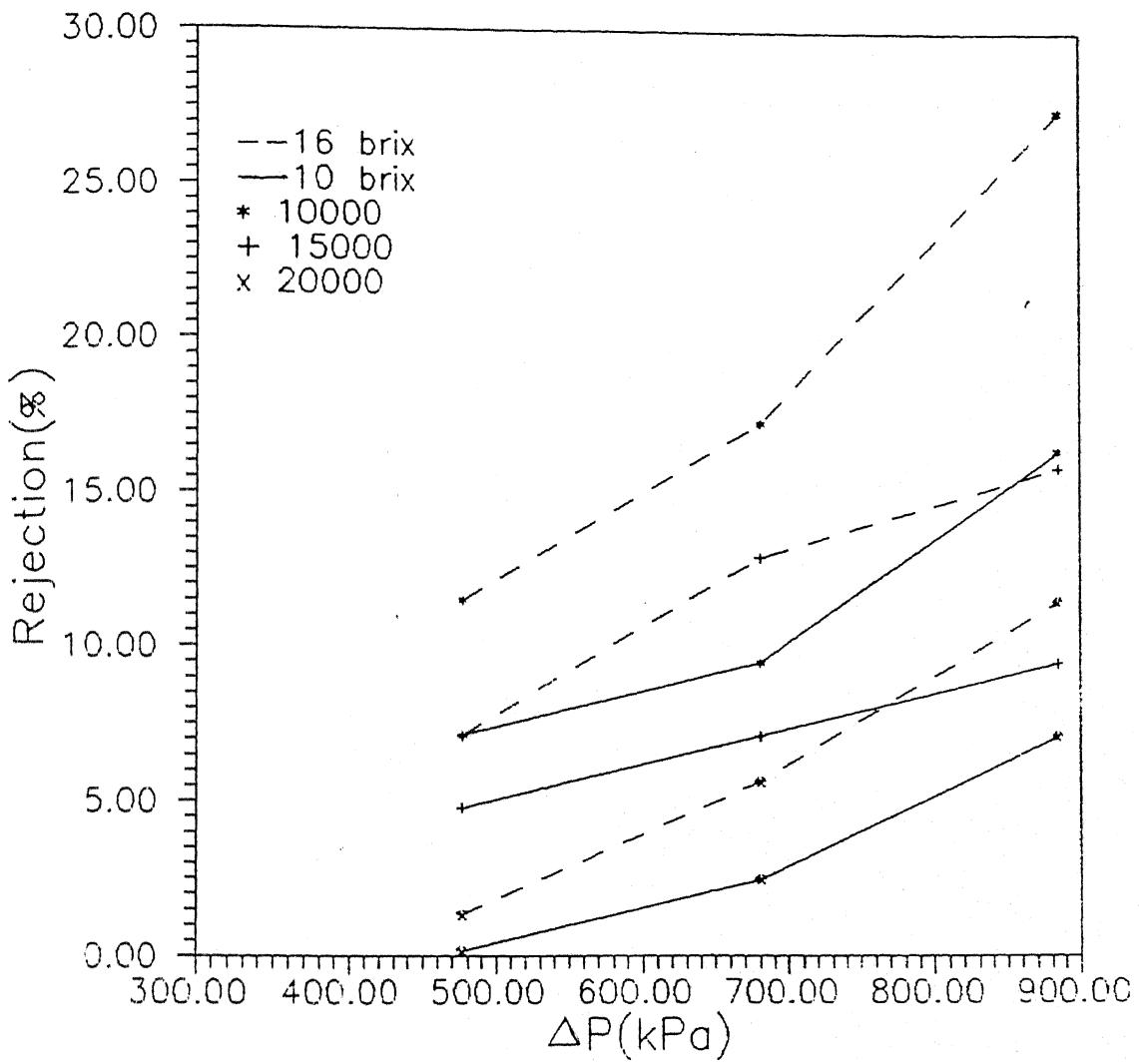


Fig 5.11 Effect of pressure on sucrose rejection as a function of bulk concentration

<b>4. Experimental work</b>	24
4.1 Instrument and material	24
4.2 Experimental device	26
4.3 Experimental procedure	29
<b>5. Result and discussion</b>	31
5.1 Pretreatment of raw sugarcane juice	31
5.2 Ultrafiltration of pretreated cane juice	32
5.3 Prediction of limiting flux	50
5.4 Comparative analysis of results	58
<b>6. Conclusions</b>	66
<b>7. Recommendation</b>	67
<b>8. References</b>	68
<b>Appendix A</b>	72
<b>Appendix B</b>	75
<b>Appendix C</b>	76
<b>Appendix D</b>	78

detail in chapter 3 . Eqn. 3.20 was used to find the specific cake resistance. Experimental steady state flux value was fed into the equation for each run and left hand term of the eqn. was plotted against time which yielded a straight line for almost all the runs. In figure 5.18, one such typical plot has been shown to prove the validity of the eqn. with our experimental data. The slope of the curve gives  $\alpha$ , the specific resistance of the cake. Figure 5.19 shows the variation of the specific resistance of cake with pressure as function of membrane cut-off at 16 brix. It shows that for lower cut-off membranes there is not much change in specific resistance at low pressures (476 kPa, 680 kPa) while at high pressure (884 kPa) there is a significant rise. However, it is evident that in 20000 membrane there is slow increase in  $\alpha$  with pressure. The rise in  $\alpha$  with pressure can be attributed to the fact that at high pressure the compaction of cake occurs thus increasing the resistance per unit mass. Furthur, it is shown in figure 5.19 that individual specific resistances increase as the cut-off size of the membrane increases which is similar to the trend already observed using osmotic pressure polarized layer resistance (figures 5.14 and 5.15). The reason of this may be explained similarly as the pore size increases permeation of LMW increases, rejecting high molecules and hence interactions of bigger molecules increases and in turn they form cake like substances which increase the resistance. Where as in the low cut-offs because of the rejection of LMW solutes as well, the larger molecules do not get much chance to interact (thus unable to form bigger macromolecules) and hence diffuse back to bulk, there by lowering the resistance.

Figure 5.20 has been plotted to depict the variation of specific resistance as ratio to  $R_m$  with respect to operating pressure and bulk concentration. It was plotted essentially to depict the effect of membrane resistance. It is evident from this figure that 15000 membrane depicts the highest  $\alpha/R_{ma}$ . This was also observed in figure 5.17 in case of osmotic pressure polarized layer model, reasons for which have already been stated there.

## 5.4 Comparative Analysis of Results

Results can be compared with respect to the obtained values of steady state flux, rejection of sucrose, polarized layer resistance and specific cake

## List of Figures

	Page no.
<b>Fig 1.1</b> A block diagram for the cane sugar industrial process	2
<b>Fig 1.2</b> A emerging flow sheet for juice clarification in sugar mills	4
<b>Fig 3.1</b> Boundary layer model for batch cell filtration	15
<b>Fig 3.2</b> concentration profile and mass balance in gel and film layer	17
<b>Fig 4.1</b> A schematic diagram of the experimental set up	27
<b>Fig 4.2</b> Ultrafiltration cell	28
<b>Fig 5.1</b> Concentration Vs refractive index calibration curve	32
<b>Fig 5.2</b> Effect of pressure on permeate flux for a 10000 membrane	37
<b>Fig 5.3</b> Effect of pressure on permeate flux for a 15000 membrane	38
<b>Fig 5.4</b> Effect of pressure on permeate flux for a 20000 membrane	39
<b>Fig 5.5</b> The comparative effect of membranes on flux as a function of pressure	40
<b>Fig 5.6</b> Effect of bulk concentration on permeate flux for 10000 membrane	42

## REFERENCES

1. V. Gekas and G. Tragardh, Membrane technology in the sugar industry, *Desalination*, **69** (1988) 9 - 17.
2. W.K. Nielsen, S. Kriestein and R. F. Madsen, Prospects and possibilities in the application of membrane filtration systems within the beet and sugar industry, *Sugar Technol. Rev.*, **9** (1982) 59 - 117.
3. V. Gekas, B. Hallstrom and G. Tragardh, Food and dairy applications : the state of the art, *Desalination*, **53** (1985) 95 - 127.
4. S. Kishihara, S. Fujii and M. Komoto, Ultrafiltration of cane juice - influence of flux and quality of permeate, *Int. Sugar J.*, **83** (1981) 35 - 39.
5. R. F. Madsen, Application of ultrafiltration and reverse osmosis to cane juice, *Int. Sugar J.*, **75** (1973) 163 - 167.
6. M. Takara and S. Nakamura, Membrane permeability of cane juice on ultrafiltration with several kinds of membranes, *Agri. Geol. Chem.*, **50** (4)(1986) 833 - 838.
7. M. Takara and S. Nakamura, Membrane permeability of ultrafiltered cane juice, *J. Agri. Chem. Soc. Japan*, **59** (8)(1985) 779 - 786.
8. S. Kishihara, S. Fujii and M. Kamoto, Improvement of flux in UF of cane juice, *Int. Sugar J.*, **85** (1012)(1983) 99 - 101.
9. S. Kishihara, H. Tamaki, S. Fujii and M. Kamoto, Clarification of technical sugar solutions through dynamic layer formed on a porous ceramic membrane, *J. Membrane Science*, **41** (1989) 103 - 114.
10. R. Kumar, V. Singh, S. Rastogi, P. Brar, R. N. Mehta and P. Raina, Initial trials with ED systems for dimineralisation of clear juice, *Proceedings of STAI Conference*, (1992).
11. M. V. J. Sharma, V. C. Malshe and J. K. Gehlawat, Studies in UF of sugar cane juice, *Chemcon, UDCT, Bombay*, (1993).
12. M. N. Patel, The potential of membrane processes in the cane sugar industry, *Proceedings of Annual Congress of South African Sugar Technologist*.
13. S. Sourirajan, Reverse osmosis separation and concentration of sucrose in aqueous solutions using porous acetate membranes, *I and EC Process Design Developement*, (1967) 150 - 160.
14. Sujoy Chattopadhyay, Removal of  $\text{Ca}^{++}$  ions from sugar solution through ED, *M. Tech Thesis, IIT Kanpur*, (1994).
15. M. V. J. Sarma, Mass transfer studies for the sugar solution concentration through reverse osmosis, *M. Tech Thesis, IIT Kanpur*, (1994).

<b>Fig 5.7</b>	Effect of bulk concentration on permeate for 15000 membrane	43
<b>Fig 5.8</b>	Effect of bulk concentration on permeate for 20000 membrane	44
<b>Fig 5.9</b>	The comparative effect of membr- ane on flux as a function of bu- lk concentration	45
<b>Fig 5.10</b>	Comparison of total rejection and sucrose rejection	47
<b>Fig 5.11</b>	Effect of pressure on sucrose rejection as a function of bu- lk concentration	48
<b>Fig 5.12</b>	Effect of bulk concentration on sucrose rejection	49
<b>Fig 5.13</b>	Comparison of calculated and predicted flux for 15000 MWCO membrane	52
<b>Fig 5.14</b>	Variation of polarised layer resistance with time for diff- erent membranes	53
<b>Fig 5.15</b>	Variation of polarised layer resistance as a function of pressure with time for 10000 membrane for 15 brix	54
<b>Fig 5.16</b>	Effect of pressure on polari- sed layer resistance	55
<b>Fig 5.17</b>	Effect of bulk concentration on polarised layer resistance	56
<b>Fig 5.18</b>	Specific resistance of cake for a 20000 membrane	57
<b>Fig 5.19</b>	Effect of pressure on specific resistance	59
<b>Fig 5.20</b>	Effect of pressure on the ratio of specific resistance and hy- draulic resistance	60
<b>Fig C.1</b>	A block diagram for algorithm	77

# APPENDIX D

MEMBRANE 10000

RI(F) = 10 brix

$$P = 476.19 \text{ kPa}$$

$$Rm = 3.965e13 /m$$

$$RI(P) = 8.0$$

$$t(s) \quad Flux(m/s)$$

$$(x10e-2) \quad (x10e6)$$

$$4.5 \quad 6.06$$

$$7.07 \quad 5.40$$

$$18.64 \quad 5.36$$

$$21.55 \quad 5.18$$

$$27.59 \quad 5.01$$

$$30.60 \quad 5.01$$

$$36.70 \quad 4.71$$

$$P = 680.272 \text{ kPa}$$

$$Rm = 4.01e13 /m$$

$$RI(P) = 7.8$$

$$t(s) \quad Flux(m/s)$$

$$(x10e-2) \quad (x10e6)$$

$$4.32 \quad 6.286$$

$$6.99 \quad 5.64$$

$$15.86 \quad 5.60$$

$$18.57 \quad 5.56$$

$$23.35 \quad 5.52$$

$$28.60 \quad 5.43$$

$$P = 884.353 \text{ kPa}$$

$$Rm = 3.965e13 /m$$

$$RI(P) = 7.2$$

$$t(s) \quad Flux(m/s)$$

$$(x10e-2) \quad (x10e6)$$

$$2.13 \quad 7.85$$

$$4.29 \quad 6.97$$

$$17.00 \quad 6.30$$

$$26.40 \quad 6.20$$

$$28.91 \quad 6.15$$

$$33.90 \quad 6.04$$

$$36.40 \quad 6.03$$

RI(F) = 13 brix

$$P = 476.19 \text{ kPa}$$

$$Rm = 3.712e13 /m$$

$$RI(P) = 11.0$$

$$t(s) \quad Flux(m/s)$$

$$(x10e-2) \quad (x10e6)$$

$$0.0 \quad 5.89$$

$$2.74 \quad 5.73$$

$$6.43 \quad 4.87$$

$$13.05 \quad 4.65$$

$$15.95 \quad 4.21$$

$$19.40 \quad 3.89$$

$$24.52 \quad 3.78$$

$$27.31 \quad 3.77$$

$$P = 680.272 \text{ kPa}$$

$$Rm = 3.960e13 /m$$

$$RI(P) = 9.6$$

$$t(s) \quad Flux(m/s)$$

$$(x10e-2) \quad (x10e6)$$

$$0.0 \quad 6.78$$

$$4.53 \quad 6.72$$

$$6.73 \quad 5.98$$

$$10.67 \quad 5.85$$

$$12.71 \quad 5.73$$

$$17.25 \quad 5.55$$

$$22.05 \quad 5.13$$

$$24.44 \quad 5.01$$

$$27.05 \quad 4.85$$

$$P = 884.353 \text{ kPa}$$

$$Rm = 4.04e13 /m$$

$$RI(P) = 8.2$$

$$t(s) \quad Flux(m/s)$$

$$(x10e-2) \quad (x10e6)$$

$$0.0 \quad 7.75$$

$$2.93 \quad 7.70$$

$$6.41 \quad 6.97$$

$$9.27 \quad 6.45$$

$$12.79 \quad 6.40$$

$$16.48 \quad 6.12$$

$$23.93 \quad 6.03$$

$$31.43 \quad 5.97$$

RI(F) = 16 brix

$$P = 476.19 \text{ kPa}$$

$$Rm = 6.77e13 /m$$

$$RI(P) = 12.2$$

$$t(s) \quad Flux(m/s)$$

$$(x10e-2) \quad (x10e6)$$

$$0.0 \quad 4.90$$

$$5.8 \quad 4.01$$

$$7.75 \quad 3.86$$

$$10.10 \quad 3.85$$

$$13.64 \quad 3.83$$

$$15.64 \quad 3.75$$

$$28.20 \quad 3.64$$

$$33.06 \quad 3.56$$

$$37.45 \quad 3.34$$

$$38.76 \quad 3.28$$

$$P = 680.170 \text{ kPa}$$

$$Rm = 5.82e13 /m$$

$$RI(P) = 11.4$$

$$t(s) \quad Flux(m/s)$$

$$(x10e-2) \quad (x10e6)$$

$$0.0 \quad 6.18$$

$$5.35 \quad 5.18$$

$$8.53 \quad 5.10$$

$$11.56 \quad 4.96$$

$$14.68 \quad 4.88$$

$$24.48 \quad 4.58$$

$$31.20 \quad 4.51$$

$$P = 884.353 \text{ kPa}$$

$$Rm = 4.81e13 /m$$

$$RI(P) = 10.0$$

$$t(s) \quad Flux(m/s)$$

$$(x10e-2) \quad (x10e6)$$

$$0.0 \quad 7.21$$

$$6.37 \quad 6.94$$

$$8.61 \quad 6.73$$

$$10.96 \quad 6.41$$

$$13.04 \quad 6.18$$

$$15.93 \quad 5.96$$

$$18.43 \quad 6.03$$

$$20.95 \quad 5.98$$

$$23.57 \quad 5.75$$

$$28.78 \quad 5.56$$

$$31.62 \quad 5.31$$

## List of Tables

	page no.
<b>Table 2.1</b> Composition of raw cane juice	7
<b>Table 5.1</b> Refractive index of limed juice before and after filtration through packed bed	34
<b>Table 5.2</b> Refractive index of limed juice before and after filtration packed bed and filter paper	34
<b>Table 5.3</b> Generated composition of limed juice	36
<b>Table 5.4</b> Tabulated results of 10000 MWCO membrane	61
<b>Table 5.5</b> Tabulated results of a 15000 MWCO membrane	62
<b>Table 5.6</b> Tabulated results of a 20000 MWCO membrane	63
<b>Table A.1</b> Refractive index - Density data for limed cane juice	73
<b>Table A.2</b> Refractive index - Viscosity data for limed cane juice	73
<b>Table A.3</b> Refractive index-Concentration data for limed cane juice	74
<b>Table A.4</b> Refractive index-Concentration data for raw cane juice	74
<b>Table B.1</b> Refractive indices of various solutes and their solutions	75

## Nomenclature

<b>a</b>	Pore Radius	m
<b>A</b>	Area of the membrane	$m^2$
<b>C,c</b>	Solute concentration	brix
<b>D</b>	Diffusivity	$m^2/s$
<b>ED</b>	Electrodialysis	
<b>J</b>	Permeate flux	$m^3/m^2/s$
$J_s$	Steady state flux	$m^3/m^2/s$
<b>k</b>	Mass transfer coeff icient	$m/s$
<b>M</b>	Molecular weight	daltons
<b>MF</b>	Microfiltration	
<b>MWCO</b>	Molecular weight cut off	
<b>NF</b>	nanofiltration	
<b>P</b>	Pressure	$N/m^2$
$R_c$	Cake resistance	$m^{-1}$
$R_m$	Membrane resistance	$m^{-1}$
$R_o$	Observed rejection	
<b>RI,<math>n_D</math></b>	Refractive index	
<b>RO</b>	Reverse Osmosis	
$R_p$	Polarised layer resistance	$m^{-1}$
$R_r$	Real rejection	
<b>t</b>	Time	sec
<b>UF</b>	Ultrfiltration	
<b>v</b>	volume	$m^3$
<b>x</b>	Distance in the film layer	m

## Greek Symbols

$\alpha$	Specific Resistance	m/kg
$\mu$	Viscosity	$kgm^{-1}s^{-1}$
$\nu$	Kinematic viscosity	$m^2s^{-1}$
$\omega$	Angular Velocity	rpm
$\pi$	Osmotic pressure	$N/m^2$
$\rho$	Density	$kg/m^3$
$\Delta$	Difference	
$\delta$	Membrane thickness	m
$\tau$	Totuosity factor	
$\epsilon$	Membrane porosity	

## Subscripts

$a$	adsorption
$b$	bulk
$c$	cake
$F$	feed
$m$	membrane
$p$	permeate
$s$	steady state, feed solution, sucrose
$w$	water

## Abstract

Ultrafiltration (UF) of limed sugarcane juice was carried out to study the flux decline behaviour and the effect on rejection of sucrose. Two theoretical models viz. osmotic pressure polarised model and cake filtration model were used to predict the resistances of the polarised layer and cake layer, respectively.

A comparative analysis of three UF membranes (viz. 10000, 15000 and 20000 molecular weight cut-off (MWCO) ) was done at different pressures (viz. 476, 680 and 884 kPa ) and three different feed concentrations (viz. 10, 13 and 16 brix ). The speed of stirrer was kept constant at 600 rpm. The models were used to furthur help in the analysis and isolate a suitable membrane among the three with respect to flux, rejection and resistance. It was observed that the permeate in all the cases was clear, transparent and slight yellow in colour. The colour diminished with the decrease in the concentration of the permeate. From the comparative analysis it was found that 15000 MWCO ( commercial grade polyamide ) membrane at 680 kPa and 10 brix yielded a considerably high flux, low sucrose rejection and low resistances. However, it was the 20000 MWCO ( laboratory grade cellulose acetate ) membrane that gave favourable results for feed ultrafiltered at high pressure and high concentration.

# **Chapter 1**

## **INTRODUCTION**

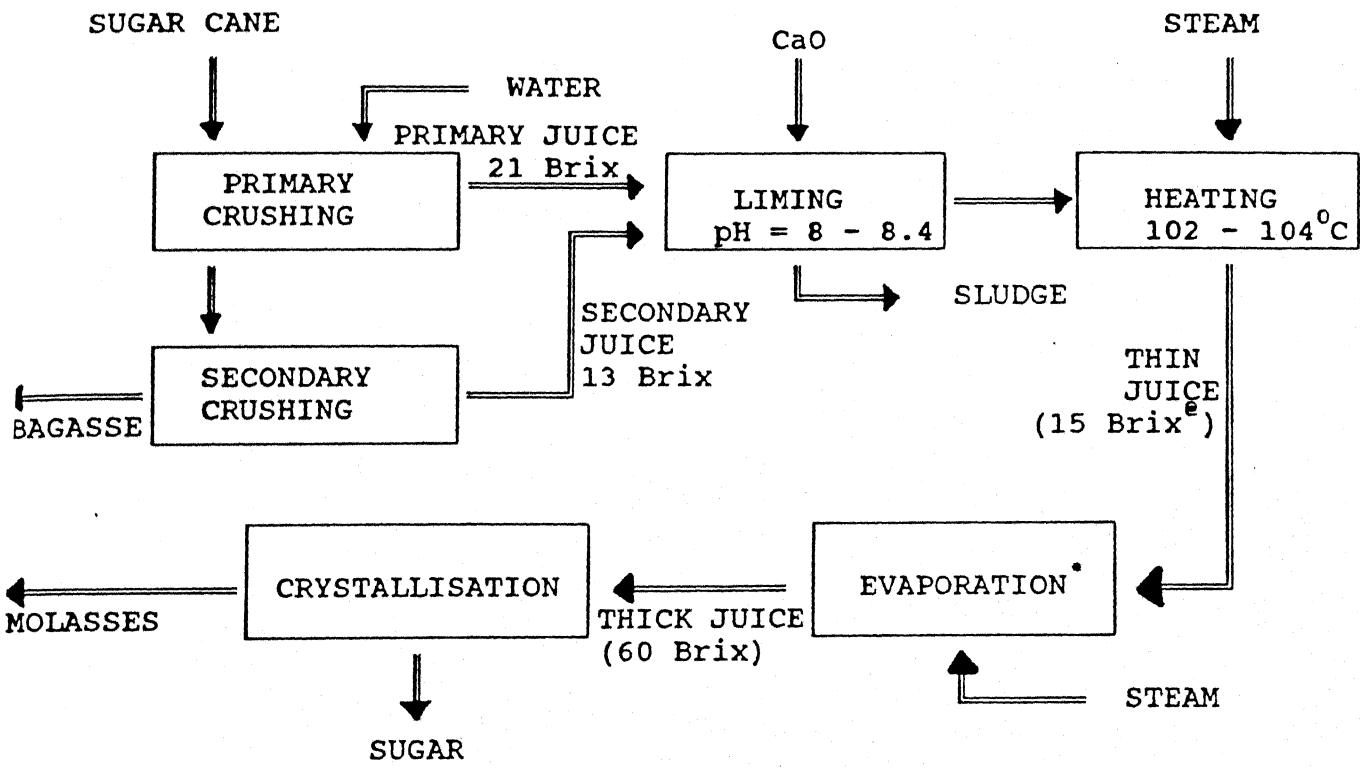
Sugar industry in India enjoys the status as one of the largest food industries. Undeniably, it is the second largest agro-based industry at present reckoning. Sugar production has exceeded 15 million tonnes mark per year during the last two years. The achievement of the industry has ranked India as one of the largest sugar producing countries of the world.

Sugar industry uses the conventional process to manufacture sugar. A block diagram of the process is shown in figure 1.1 [1].

The raw juice obtained after crushing sugarcanes contains sucrose along with various impurities which includes colloidal, suspended and colouring matter. Therefore, the juice is clarified by liming, followed by heating and separation of the subsequently formed sludge. More sophisticated clarification methods like affination, active carbon treatment, phosphatation, etc. are employed in refined sugar processing. After clarification, juice is concentrated and finally sugar is crystallised out in crystallisers, leaving behind a thick syrup, known as molasses.

The conventional process has been used since years without any significant modifications, despite of the fact that it is highly intensive. Besides, it has many other drawbacks. Some of them are:

1. Incomplete removal of high molecular weight substances during clarification which is more oriented towards colour elimination.
2. High doses of chemical additives increase the percentage of inorganic impurities, which in turn poses massive problems like scaling of evaporators and retardation of sugar yield.



Concentration of sucrose in wt%

Evaporation is done in Multi-Effect Evaporators

Fig 1.1 A block diagram for the cane sugar industrial process

3. Inversion of sucrose in alkaline medium leads to large amount of molasses formed.

4. Concentration of juice by evaporators and condensation of same consumes enormous energy.

Increasing cost of energy and ever rising demand for quality sugar has compelled the scientist to look for alternative efficient routes. Application of integrated membrane technology is one such step in this direction.

Membrane technology include Reverse Osmosis(RO), Nanofiltration(NF), Ultrafiltration(UF), Microfiltration(MF) and Electrodialysis(ED) separation techniques. These are not only energy-efficient and cost-effective but are also environmental friendly. Hence, offer a distinct advantage in clarification and concentration of multi-component solutions and suspensions.

A brief review of the pioneering studies on the application of the membrane systems in sugar industry was compiled by Madsen et al [2], Kishihara et al [4] and Gekas et al [1,3]. These reports suggest the following streams for application of membrane techniques:

1. The clarification of raw juice, after liming by UF.
2. The treatment of thick juice, after evaporation by UF.
3. Treatment of molasses by ED or UF.
4. Treatment of raw sugar by UF.

The first of the four applications has been most studied. It is because UF of clarified limed juice offers following advantages:

1. Completion of purification by removal of high molecular weight substances like dextrans, waxes, etc.
2. Reduction of the volume of juice prior to evaporation making this step less costly and evaporators, free of scaling problems.
3. High final quality product.

However, other applications are also studied but to a lesser extent. The hindering factors being high viscosity of thick juice and raw sugar and changing properties of molasses, that offer processing problems. Though membrane systems are finding application in industries in a big way but the major drawback of flux decline with time has prevented them from being accepted commercially. The decline in flux is due to the phenomenon of concentration polarization which leads to fouling of the membrane and its short life span. These phenomena are discussed in detail in chapter-3.

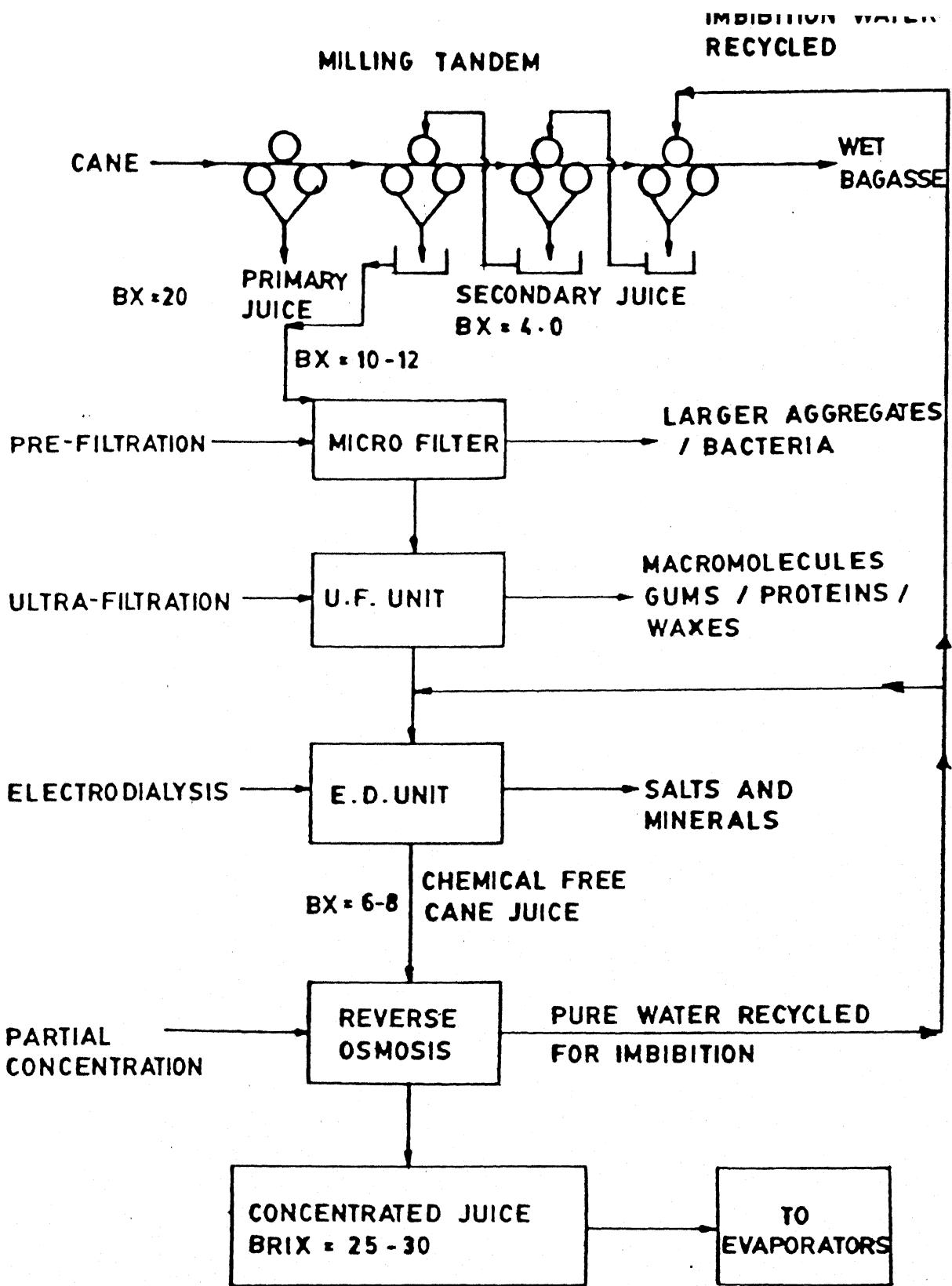


Fig 1.2 A emerging flow sheet for juice clarification in sugar mills

Several attempts have been made [1-13] to find a suitable molecular weight cut-off (MWCO) membranes that would give a clear, light coloured permeate with same sucrose content. But little or no attention has been paid to study the limiting flux phenomena. Such attempts would help in studying the effect of influencing parameters in UF for the efficient design of a unit operation for treating cane juice to recover sugar.

The present work is a continuation of on going research [14-16] being carried out in our laboratory. Based on these research works a scheme has been developed for sugar processing using different membrane separation systems. A flow diagram of which is shown in figure 1.2.

However, the present work is concentrated on the first stage i.e, the clarification of limed cane juice using UF. The main objectives of the work were:

1. To select a cold liming pretreatment and prefiltration <sup>method</sup> to eliminate the coagulated and settled impurities.
2. To study the effect of various parameters (viz. UF membrane of different MWCO, pressure and concentration ) on permeate flux and rejections.
3. To analyse the behaviour of flux decline using different models and theories.

# **Chapter 2**

## **LITERATURE CITED**

### **2.1 Industrial Process for Sugar Manufacture**

#### **2.1.1 From Sugarcanes**

The conventional process used for sugar manufacture is illustrated diagrammatically in figure 1.1 [1]. The sugarcane is chipped, shredded and crushed to extract juice. The juice contains sucrose along with various other colloidal and suspended impurities like dextrans, proteins, starch, colouring matter, organic acids etc. It is first limed to neutral or slightly alkaline pH to coagulate a part of the colloidal matter and precipitate some other impurities. A typical composition of cane juice is given in table 2.1.[17]. The clarified juice obtained still contains impurities, mainly in colloidal form giving juice a hazy appearance, hence the quality of sugar obtained is very poor. The reason is that the clarification method mainly aims at the elimination of colour and does not to any large extent, removes macromolecular substances like dextrans, starch, fat, waxes, arabinogalactans, bacteria, etc. The mixture is then heated at 100°C and settled in clarifiers. The underflow sludge from the bottom of the clarifier is filtered to extract the remaining cane juice and the separated mud goes for the final disposal. The clarified juice is concentrated in multiple effect evaporators. The concentrated juice is now sent to vacuum pan crystallisers, where the sugar is crystallised and crystals are separated using a centrifuge. The remaining part is known as molasses. Furthur,

Constituents	Wet basis (%)	Constituents	Wet Basis(%)
Water	74.5		74.5
Ash	0.5	silica, $SiO_2$	0.25
		potash, $K_2O$	0.12
		soda, $Na_2O$	0.01
		lime, $CaO$	0.02
		magnesia, $MgO$	0.01
		iron, $Fe_2O_3$	trace
		phosphoric acid, $P_2O_5$	0.07
		sulfuric acid, $SO_3$	0.02
		chlorine, Cl	trace
Fibre	10.0	cellulose	5.50
		pentosans	2.00
		cane gum	0.5
		lignin bodies, etc.	2.0
Sugar	14.0	sucrose	12.5
		dextrose	0.9
		levulose	0.6
Nitrogenous bodies	0.4	albuminoids	0.12
		amids (as asparagin)	0.07
		amido acids (as aspartic)	0.20
		nitric acids	0.01
		ammonia	trace
		Xanthin bodies	trace
Fats, wax	0.2		0.20
Pectins	0.2		0.20
Free acids	0.08	malic, succinic etc.	0.08
Combined acids	0.12	malic, succinic etc.	0.12
	100		100

Table 2.1 Composition of raw cane juice.

it is sold as a by product. The sugar crystals are dried and sized for final packaging [35].

In refined sugar manufacturing, sophisticated raw juice clarification methods are applied. These include affination, phosphatation, active carbon treatment, ion exchange, resin treatment etc. depending upon the final quality of the final product desired.

### 2.1.2 From Other Sources

Sugar beet is another major source of sugar. It also has a very high sucrose content. Its advantage over cane is that it can grow well in temperate zones too, whereas cane grows only in tropical and semi-tropical climates[35].

The industrial processes is quite similar to that for cane sugar industry [1].

Raw juice is obtained by slicing the beets and by extracting the sugar from the slices in a continuous counter current process at 70-80°C. Clarifications are carried out in two steps; liming at 35°C in the first step and at 85°C in the second step. The thin juice is concentrated and the thick juice obtained is crystallized. Molasses is obtained as a by product [2] .

Though all plants produce some sugar, but only beets and canes are the major sources. Other minor sources are the maple tree, certain palm trees and honey.

Sugar is one of the most important ingredients of everyday food. It is not prized for its sweet taste alone, for it supplies 13% of the energy required for existence. As a consequence, sugar production and refining is a huge industry and every effort is being made to make the manufacture of sugar as economical as possible. It , however, is one of the oldest technique that has prevailed over many years without any significant modifications or development. In addition , it is a highly energy intensive process, using comparatively valuable fuel material which could be better used as raw material for various other industries.

A potential breakthrough in the sugar manufacture came in the year 1971 when De Danske Sukkerfabrikker [DDS] carried out a series of experiments on clarification of cane juice and the beet using semipermeable membranes [2,5] . Since then quite a lot of effort and work has been done in this direction. The scientists have now come up with the application of membrane separation techniques to the following streams in cane sugar processing [2,3] :

1. The clarification of the raw juice, after liming by MF or UF.

2. The treatment of thick juice after evaporation by UF.
3. The treatment of molasses by UF or ED.
4. The treatment of raw sugar by UF.

It is the first application which had been and have been extensively studied. Though the other applications have been studied but to a lesser extent.

## 2.2 Review Of The Membrane Systems Tested

In 1973, R.F. Madsen [5] performed experiments on a cellulose acetate UF membranes of 6000, 20000 and 40000 molecular weight cut-off (MWCO) and RO membranes of 200 MWCO using cold clarified juice(limed) of about 7 pH as feed. He observed that in most experiments the flux dropped to one forth of its initial value within two hours and the membranes had a yellowish red deposit on the surface. The deposit he thought was wax. He found from comparative test that 20000 MWCO membrane was most suitable as 40000 MWCO membrane gave a permeate with a high colour and a little turbidity. While the 6000 MWCO membrane had a high sucrose rejection. He also reported that RO could be used only if complete removal of colloidal and suspended impurities is achieved.

In 1981, Kishiara et al.[4] performed experiments to examine the effect of various pretreatment procedures on the flux of sugar cane juice during UF and came out with the following conclusions :

- Liming of the juice to pH 7.5 gave the highest fluxes. Removal of wax from the sugarcane did not improve the flux.
- The effect of temperature was very important as an increase from 30 - 60°C almost doubled the flux.
- Membranes with MWCO of 10000 - 30000 were most suitable for clarifying the limed cane juice.

In 1982, Nielsen et al.[2] introduced their investigations on the use of membrane having increased stability at extreme pH and temperature. He suggested that it should be possible to introduce membrane filtration as a unit operation in the sugar industry because the improvement in the flux can be achieved and the microbiological contamination of juice can be avoided.

In 1983, Kishiara et al.[8] carried out UF experiments on raw juice and limed juice to investigate the effects of various flocculants, celite and bagasse on the flux. The flocculants and celite had no significant effect on the raw

juice or limed juice. However, in the presence of bagasse the flux of limed juice remained at its initial value for 6 hours. In its absence the flux of limed juice decreased rapidly during early period and after 2 hours was only one third of that in the presence of bagasse. In its absence the membrane surface was covered by a brownish deposit after the experiment but in its presence no deposit was observed. The membrane subjected to the limed juice could be restored to its original capacity by wiping the surface with a sponge and treating with the alkali.

In 1985, Taka et al. [7] ultrafiltered raw cane juice through PM - 30, AM -10, YM - 5 and YM - 2 hydrophilic UF membranes. They observed that first three membranes gave high flux ( $\sim 0.8 \text{ ml cm}^{-2} \text{ min}^{-1}$ ) that further improved by previous liming at  $85^\circ\text{C}$ . In PM - 30 and AM - 10 ( 30000 and 10000 MWCO) flux diminished quickly at early periods. They reported that protein and starch contents in permeate and retentate were lowest after preliming the juice to pH 7 at  $85^\circ\text{C}$  and also rejection of sucrose was lowest ( 2.9%) using YM - 5 membrane. This membrane was considered the most suitable for limed juice while PM - 30 was most suitable for raw juice.

In 1986, [6] the same authors undertook a pilot plant study of clarified juice applied to UF membranes. They tested three membranes of 20000, 25000 and 100000 MWCO. The juice was not pretreated as three membranes rejected 100% dextran and turbidity and starch over 98%. They reported that 100000 membrane removed 50% of colour which could be due to concentration polarization. Membranes were cleaned with 0.25% NaOH at  $70^\circ\text{C}$  when flux dropped to 20% of its initial value but could not restore the original flux. They found that sugar losses were about 0.5% and concluded that UF process will be uneconomic in the present conditions.

In 1989, Kishiara et al. [9] carried out UF by means of a self rejecting membrane (SRM) formed dynamically on a ceramic tube for clarification of cane juice and other technical sugar solutions. They came out with the following results :

- Suspended substances in cane juice were completely removed.
- The purity of the juice was increased and the viscosity was decreased.
- The permeation flux was large.
- Recovery of sucrose from syrup was improved and sucrose crystals produced were thin in colour.
- The rate of crystal growth in ultrafiltrate was also large.

Though the life span of SRM formed on ceramic tube is unknown but it

is thought that it is more durable than the organic UF membranes.

In 1990, Punidades et al.[51] studied the application of cross flow MF by ceramic membranes in sugar refining. They studied the effect of pore diameter of the membrane(0.1-1.4  $\mu\text{m}$ ), start up method, average transmembrane pressure(2 - 5.5 bar), cross flow velocity (5 - 8 m/s), temperature (80 or 90°C), volume - concentration ratio (1 - 5.1) and operations with or without backflushing. They concluded that raw sugar solutions can be efficiently clarified by MF through membrane of pore diameter 0.1 - 0.5  $\mu\text{m}$ , which gave 47% decolourization and practically 100% turbidity removal. Secondly, backflushing failed to increase the flux significantly.

All the works reported above aim chiefly at the removal of macromolecular organic impurities and colour, ignoring the inorganic impurities of clear juice which pose massive problems during processing of cane juice. They are solely responsible for scaling problem in heat exchanger, retardation rate of sugar crystallisation, increase in viscosity and higher molasses formation. Therefore, the elimination of these impurities is as important as removal of organic impurities. Raina et al.[10], Sujoy[14] and Sahu[16] did some research on this line using ED demineralisation technique. From their experiments they observed that :

- Calcium removal reduces the quantity of molasses formed.
- Sugar loss can be reduced 0.05% of cane by reducing CaO content by 100 ppm.
- The colour of juice can also be reduced.

In brief the applications of various membrane separation techniques in the processing of cane juice to produce high quality sugar has been shown in figure 1.2. The advantages of these can be summarized as follows :

1. Complete removal of macromolecular organic impurities like proteins, dextrans, gums, etc. by UF.
2. Partial removal of inorganic impurities by ED.
3. Partial concentration of juice by RO.
4. Retention of aroma.
5. Prevent thermal damage of products due to low or ambient temperature of operation.
6. A superior final quality product [2,4].

Besides its varied advantages, membrane processes have to over come its severe drawback of flux decline and consequent fouling to be accepted commercially as an attractive viable alternative process for sugar manufacture.

## **2.3 Review Of Studies On the Concentration Polarisation in UF**

A summary of the flux decline due to concentration polarisation, its limitations and the ways to reduce concentration polarisation and membrane fouling are discussed in detail by Belfort et al.[36].

The dynamics of polarisation in stirred and unstirred UF cell was studied by Chudacek et al.[27]. The simple Carman-Kozeny equation was shown to be good approximate to the amount of solute in the polarised layer. Filtration theory was utilised to evaluate the specific resistance<sup>of</sup> cake.

Wijman et al.[37,38], have explained the flux limitation in the UF in terms of osmotic pressure. On the other hand Jonson [52] suggest that a degree of osmotic limitation have to be considered along with gel polarisation model. Dickson et al.[43] and Do et al.[44] have also discussed in this line. The limiting flux is independent of applied pressure and is strongly dependent on stirring and bulk concentration.

Nakao et al.[39,40] considered cake filtration theory to analyse concentration polarisation and fouling in unstirred conditions. This was followed soon by Aimar et al.[28]. Aimar et al. have illustrated four models for evaluating concentration polarisation. A model based on adsorption isotherm and another on gel layer growth have also been derived and discussed. In the same direction Chiranjib et al.[31,32] have proposed osmotic pressure model and gel layer model for predicting the resistance build up on the membrane due to retention of the solutes.

With the increase in the complexity of the problems , a large usage of numerical modelling procedures have been experienced lately and quite a few studies on mathematical modelling and simulation of membrane modules. Several works [41,42,45,46] have been reported in this respect. The present day membrane literature consist of innumerable studies on different aspects of flux decline and a number of reviews have been written on these works. Of these, Gekas and Hallstorm [47] present a detail discussion on factors like porosity, wall roughness, change in viscosity, diffusion coefficient. Belfort and Nagata [48] enumerated the governing equations for transport and adsorption phenomena in porous membrane ducts. Two recent reviews deal extensively with the studies made on flux decline and its causes in UF [49,50].

# **Chapter 3**

## **THEORETICAL CONSIDERATIONS**

### **3.1 Ultrafiltration**

Ultrfiltration (UF) is size-exclusion based pressure driven membrane separation process. The UF membranes have a pore size range from 10 to 1000 Å and are capable of retaining species in the molecular weight range of 500 to 500,000 daltons. The species it can typically reject include proteins, biomolecules, polymers and colloidal particles. The UF can be thought simply as a flow through pores in which separation is a seiving process based on relative molecular size. It can be thought of as performing following functions:

1. Feed clarifications.
2. Concentration of rejected solutes.
3. Fractionation of solute

### **3.2 Flux**

The water flux through porous membrane can be described by Darcy's law which states that flow rate is directly proportional to the applied pressure gradient:

$$J = 1/A(dv/dt) = \frac{\Delta P}{\mu_w R_m} \quad (3.1)$$

During UF of macromolecular solutes the linear relationship between  $J$  and  $\Delta p$  does not hold. The volumetric flux is then given by:

$$J = \frac{\Delta P - \Delta \pi}{\mu_s R_m} \quad (3.2)$$

where,  $R_m$  is intrinsic hydraulic resistance and  $\Delta \pi$  is difference in osmotic pressure of feed and permeate phases. Flow through pores of UF membranes is modified by factors such as pore tortuosity, pore diameter, membrane thickness and porosity. Hence, the resistance is given by:

$$R_m = \frac{8\tau\delta}{\epsilon a^2} \quad (3.3)$$

The UF membranes are plastic in nature and can yield under pressure thus making compaction a compulsory step before use of any membrane. The  $R_m$  therefore is also a function of pressure history. Membranes have to be compacted at a pressure higher than the operating pressure.

Feed (liquid) phase mass transfer resistance and resistance due to gel layer or cake formation on the membrane surface are extremely important. Hence, fouling and concentration polarization are major important effects of UF processing.

### 3.3 Concentration Polarization

The accumulation of the rejected species on the membrane surface is called concentration polarization. Because the solute is retained by the membrane hence, the concentration at the wall ( $C_m$ ) increases. The value of  $C_m$  is determined by the balance between solute brought to the surface by the convective flow of the solvent and that which diffuses back in to the bulk. Consequently, the membrane effectively experiences a higher feed side concentration resulting in reduced flux and rejection.

In UF, the macromolecular solutes and colloidal species usually have insignificant osmotic pressure. Nevertheless, flux is affected along with rejection by concentration polarization and the formation of gel layer or secondary membrane. In UF, the higher flux often drives the concentration at the wall above the solubility limit and we are accustomed to operating in a gel polarised regime (figure 3.2). In this regime, the concentration at the

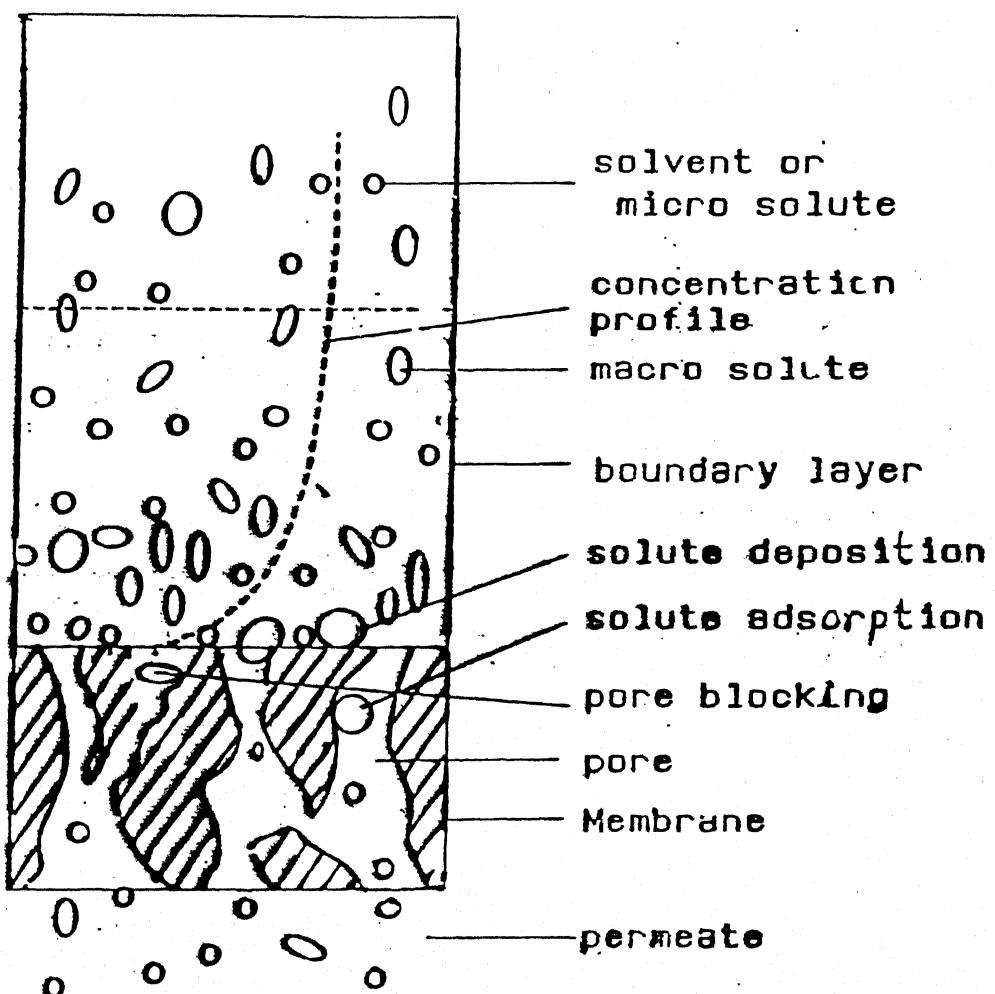
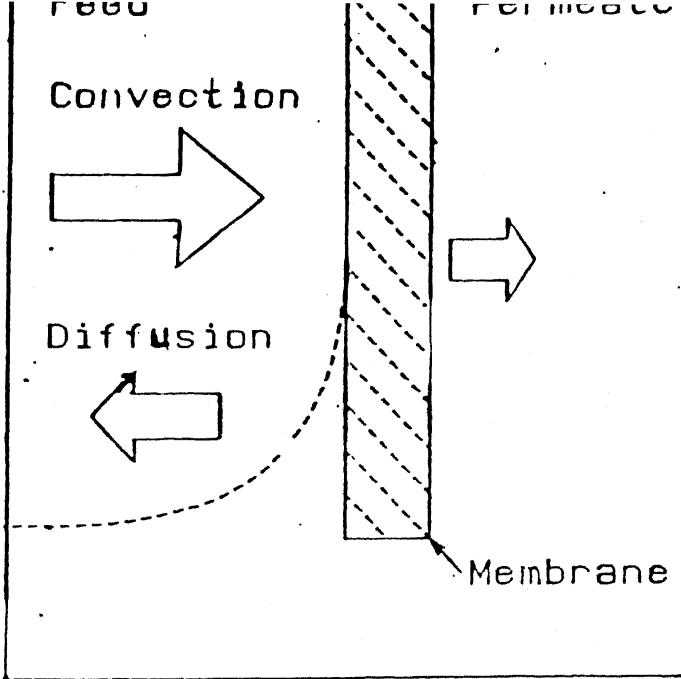


Fig 3.1 Boundary layer model for batch cell filtration

surface is fixed and solute passage occurs at a constant rate, irrespective of changes in the feed concentration. The flux, then is called limiting flux and is expressed as:

$$J = \left( \frac{\Delta P - \Delta \pi}{R_m + R_c} \right) \quad (3.4)$$

where,  $R_c$  is the resistance offered by the deposited cake.

### 3.3.1 Resistance in series

The additional resistances offered by the two layers on the membrane feed side surface - the boundary layer and a gel or cake layer are responsible for flux decline. The flux reduction phenomenon is analysed by a boundary layer model illustrated in figure 3.2. A steady state mass balance over concentration boundary layer gives:

$$J(C - C_p) = -D \left( \frac{dc}{dx} \right) \quad (3.5)$$

Integration of the equation using the following boundary conditions yield eqn. 3.6 : 1.  $C = C_b$  at  $x = \delta$  2.  $C = C_m$  at  $x = 0$

$$J = k \ln \frac{C_m - C_p}{C_b - C_p} \quad (3.6)$$

where,  $k = D/\delta$ , is the mass transfer coefficient.

For stirred cell,  $k$  can be found from the following correlation[24]:

1. Laminar boundary layer over membrane surface

$$\frac{k_s r}{D_s} = 0.285 \left( \frac{\omega r^2}{\gamma} \right)^{0.55} \left( \frac{\gamma}{D_s} \right)^{0.33} \quad (3.7)$$

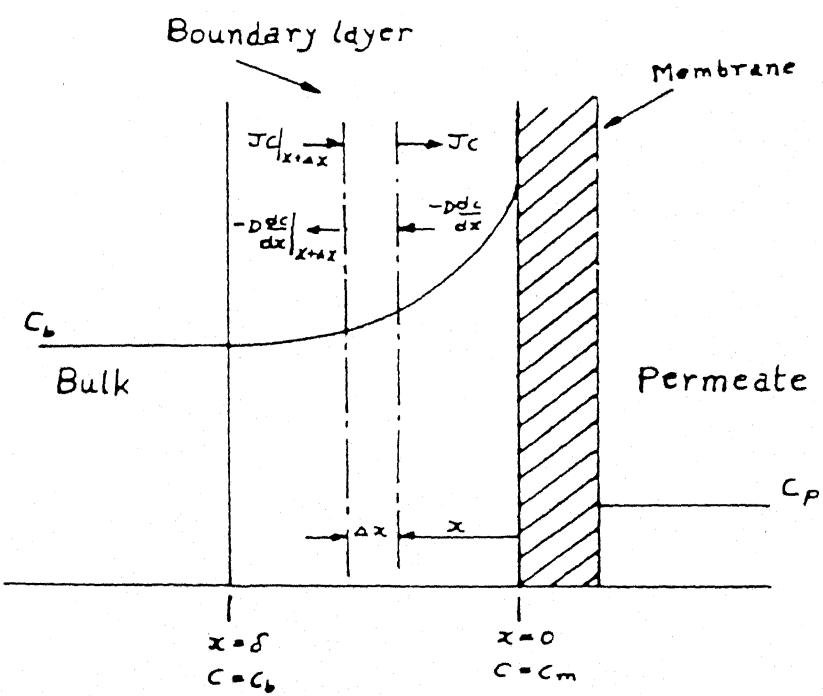
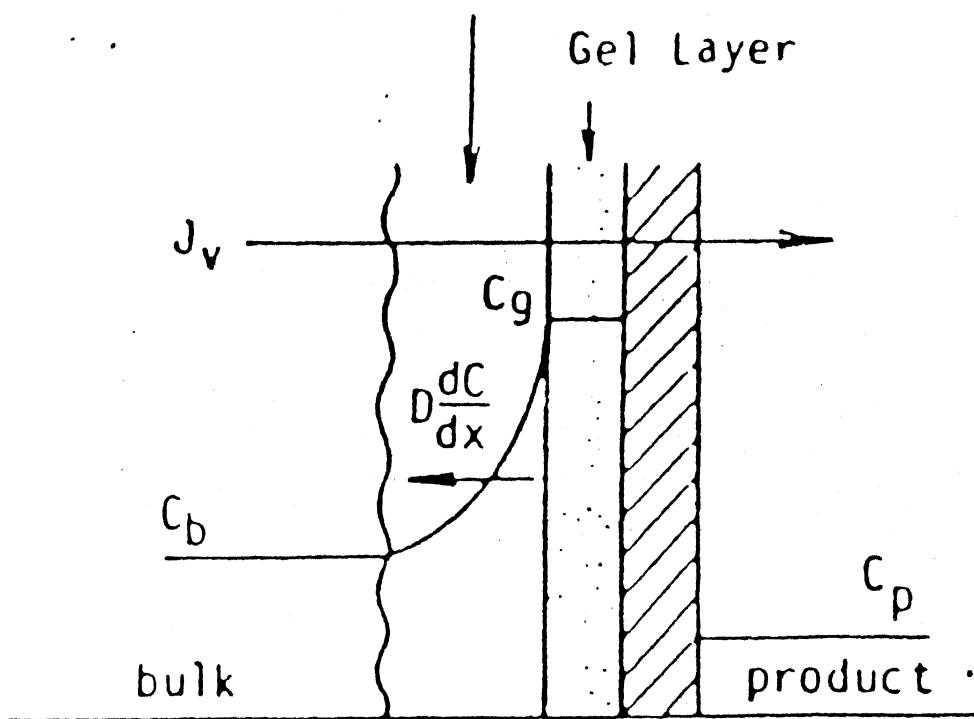
when,

$$8000 < \left( \frac{\omega r^2}{\gamma} \right) < 32000 \quad (3.8)$$

2. Turbulent boundary layer over the membrane surface.

$$\frac{k_s r}{D_s} = 0.0443 \left( \frac{\omega r^2}{\gamma} \right)^{0.75} \left( \frac{\gamma}{D_s} \right)^{0.33} \quad (3.9)$$

## Boundary Layer



**Fig 3.2** Concentration profile and mass balance in gel and film layer

when,  $32000 < \left(\frac{\omega r^2}{\gamma}\right) < 82000$

where,  $r$  = cell radius, cm

$\omega$  = stirrer speed, rad/sec

$\nu$  = kinematic viscosity,  $\text{cm}^2/\text{sec}$

$D$  = diffusivity of solute,  $\text{cm}^2/\text{sec}$

### 3.3.2 Fouling

It is defined as the additional resistance offered by the deposition of solutes on the surface or within the pores (adsorption), figure 3.2 account for these resistances, eqn.3.2 is modified as:

$$J = \frac{\Delta P - (\pi_w - \pi_p)}{\mu_s(R_m + R_a + R_p)} \quad (3.10)$$

where  $R_a$  corresponds to the resistance due to adsorption and  $R_p$  is due to accumulation. Increase in applied pressure causes densification of the cake or gel layer and therefore cancels the expected flux increase. Secondly the solute adsorbed in the pores of the membrane can either completely block them or effectively reduce their diameter. As a result of either of these mechanisms, the flux through membrane is reduced while the rejection may essentially be constant or may increase. Fouling is of two types :

1. **Reversible Fouling** : It refers to fouling that can be reversed by cleaning it with water or some other cleaning agent.

2. **Irreversible Fouling** : It is the main reason that necessitates membrane replacement.

### 3.4 Rejection

It reflects the ability of a membrane to retain certain species. It is defined in two ways as :

1. **Observed Rejection** : It is based on the bulk concentration of the feed and is given by :

$$R_o = 1 - \frac{C_p}{C_b} \quad (3.11)$$

**2. Real Rejection :** Due to concentration polarization the concentration of species <sup>at the surface</sup> is much higher than the bulk hence, it is based on membrane surface concentration and is defined as

$$R_r = 1 - \frac{C_p}{C_m} \quad (3.12)$$

### 3.5 Molecular Weight Cut-off Profile

The rejection characteristics of UF membranes are usually expressed as nominal molecular weight cut-off (MWCO). This number actually refers to the molecular weight (daltons) of a species, which would be expected to have  $R_o$  (eqn. 3.10) value of atleast 0.9. A MWCO profile or retention curve is constructed by measuring the  $R_o$  values of chemically similar compounds of varying molecular weights . There are two types of MWCO UF membranes. They are :

1. **Sharp Cut-off Membrane :** It is microporous ultrafilter in which the width of the pore size distribution is so small such that the size difference between the solute which is completely retained and a solute which passes through the pores unhindered is small.
2. **Diffuse Cut-off Membrane :** In such membranes there is wide pore size distribution.

### 3.6 Estimation Of Additional Resistance Offered By The Membrane Due To Retention of Solutes

#### 3.6.1 Osmotic Pressure Polarized Layer Model

The model [Winston] explains the flux reduction by the increase in osmotic pressure difference from the feed / membrane surface to the permeate phase that has to be overcomed. In the case when large molecular weight species accumulate at the membrane surface with back diffusion in to the bulk, the osmotic pressure difference  $[\pi_w(C_w) - \pi_p(C_p)]$  may be significant. The concentration polarization model eqn.3.4 along with the basic osmotic pressure

model gives the following equation as a function of time :

$$J(t) = \frac{\Delta P - \Delta \pi}{\mu_s [R_m + R_a + R_p(t)]} \quad (3.13)$$

This is used to account and estimate for the polarized layer resistance,  $R_p$  as a function of time. An iterative technique was adopted to obtain membrane surface concentration ( $C_m$ ), mass transfer coefficient ( $k$ ) and polarized resistance  $R_p$ . The mass transfer coefficient  $k$  was calculated from eqn.3.7 or eqn.3.8 depending on the Reynold's number. The physical properties that is density and viscosity were determined at the average concentration prevailing the boundary layer. The average concentration was obtained by making a differential mass balance over the element of thickness  $\Delta x$  shown in figure 3.2. At steady state :

$$JC|_{x+\Delta x} - JC|_x + D[\frac{dc}{dx}|_{x+\Delta x} - \frac{dc}{dx}|_x] = 0 \quad (3.14)$$

or

$$J \frac{dc}{dx} + D \frac{d^2c}{dx^2} = 0 \quad (3.15)$$

This equation is solved under the following boundary conditions:

1. at  $x = 0, C = C_m$
2. at  $x = \delta, C = C_b$

Assuming  $D$  to be independent of concentration, the solution of this equation is obtained as:

$$C = C_m - (C_m - C_b) \left[ \frac{1 - e^{-Ax}}{1 - e^{-A\delta}} \right] \quad (3.16)$$

The average concentration prevailing over the boundary layer is defined as :

$$\bar{C} = \frac{1}{\delta} \int_0^\delta C dx \quad (3.17)$$

Substituting eqn.3.13 in eqn.3.14, the average concentration is obtained as:

$$\bar{C} = C_m - (C_m - C_b) \left[ \frac{1}{1 - e^{-J/k}} - \frac{k}{J} \right] \quad (3.18)$$

### 3.6.2 Cake Filtration Model

In unstirred UF, solute is carried convectively to the membrane surface where it accumulates and provides an increasing barrier to solvent flow. Solute diffusion back from the membrane moderates this effect by reducing the transport of solute to the membrane. During the initial stages convective transport predominates, and by neglecting diffusional back transport we can analyse unstirred UF in terms of conventional pressure filtration theory [26]. The assumption made was that the polarised concentration profile is established very rapidly so that solute starts to deposit cake or gel instantaneously, where the concentration is constant and equal to  $C_g$  [27].

It is also assumed that the amount of particles deposited by convection is proportional to their bulk concentration,  $C_b$  and to the volume filtered  $v$  [29]. The eqn.3.4 can be written as :

$$J = \frac{\Delta P - \Delta \pi}{\mu[R_m + \alpha C_b v]} \quad (3.19)$$

where  $\alpha$  is the specific resistance of the cake. For stirred UF cell [28], the effect of back diffusion cannot be neglected during the initial polarisation period since it could be substantial. In this case, the convective flux of matter is partially balanced by a constant removal flux,  $J_s$ . Such a model has been proposed by Kimura et al. [30] for fouling reverse osmosis membrane and later by Fane et al. [27] in differential form.

In the gel model the back diffusive flux,  $J_s$  is constant and equal to  $k \ln(\frac{C_g}{C_b})$ . The gel then grows until the permeation rate is equal to  $J_s$ , that is the flux at steady state, eqn 3.19 can then be written as follows:

$$\frac{dv}{dt} = J = \frac{\Delta P - \Delta \pi}{\mu[R_h + \alpha C_{bp}(v - J_s t)]} \quad (3.20)$$

where  $C_{bp} = C_b - C_p$ . On integration of eqn. 3.20 we obtain the following equation:

$$J_s \ln\left[\frac{J(t)}{J_s - J(t)}\right] - \frac{J_s}{J(t)} = \frac{\alpha C_{bp} J_s^2 \mu t}{\Delta P - \Delta \pi} - \frac{J_s}{J(0)} + J_s \ln\left(\frac{J(0)}{J_s - J(0)}\right) \quad (3.21)$$

It is necessary to know the value for  $J_s$  in order to check the validity of this model. When  $J_s$  is known, plotting the left hand term of eqn.3.18

versus  $t$  should give a straight line. On the other hand, if the constant wall concentration assumption is not valid, the value of the back diffusion flux is no longer constant and eqn.3.18 does not describe experimental results.

## 3.7 Ultrafiltration Of Cane Juice

### 3.7.1 Analysis of Cane Juice

The raw cane juice was obtained from a local vendor. The composition of the juice varied with place and season. Even crop from one place grown in the same season yield sugarcane juice of varying composition. Hence, it is extremely difficult to analyse the composition of raw juice everyday, before use. Therefore, composition of raw cane juice available from the literature [17] was utilised for the present work. The composition of raw juice is shown in table 9.1. However, no data could be found for composition of the limed juice. Hence, a theoretical table was generated based on the characteristics of the constituents of juice. The solutes with molecular weight less than 10,000 are grouped under permeable solutes while those with molecular weight greater than 10,000 are grouped under non-permeable solutes. The approximate theoretical table so developed is shown in *table 5.3*.

### 3.7.2 Estimation Of Rejection

The concentration of the feed and the permeate is estimated by measuring refractive index (RI). Therefore, the rejection of solutes can be mathematically expressed as:

$$R = \frac{RI_F - RI_p}{RI_F} \quad (3.22)$$

Here  $R$  gives total rejection of solids not of an individual specie. Hence, to find rejection of an individual solute, the measured RI is multiplied by the weight fraction of the solute in the feed and permeate, respectively. For example, to find the rejection of sucrose eqn.3.22 is modified as :

$$R_s = \frac{RI_F \times 0.71 - RI_p \times 0.83}{RI_F \times 0.71} \quad (3.23)$$

Similarly, for each permeable solutes, this rejection can be determined. Here it has to be noted that the rejection of the non-permeable solutes have been assumed to be 100%.

# **Chapter 4**

## **EXPERIMENTAL WORK**

### **4.1 Instruments and Materials**

#### **Membrane**

Type	spectra/por;Spectrum Medical Industries,USA polyamide/comm;Hydraunautics Ltd,India
Nature	Flat,disk,assymrtric,anisotropic hydrophillic
Diameter	76 mm
MWCO	10000,15000,20000
Composition	Cellulose acetate polyamide
pH	2 to 10
Allowable max temp.	90°C
Support	Highly porous, 150 to 300 $\mu\text{m}$
Skin	0.1 to 0.5 $\mu\text{m}$
Sterilization	By hydrogen peroxide or formalic or ethylene oxide
Suggested cleaning	By detergent with enzyme or 2.5% hydrogen peroxide. 1 to 2% formaldehyde
Storage	1 to 2 % formaldehyde

**Stirrer**

Diameter of the stirrer	56 mm
Type of packing used in the gland	Asbestos packing
Sealing fluid	Sillicone grease

**Stirrer motor**

Type	Shunt, continuous rating
Supply	220 v. DC
Ampirage	0.85 amp
Horse power	0.125 Hp
rpm	1450

**Pump**

Type	Reciprocating
Supply	220 v, 1 phase, 50 Hz
Ampirage	2.6 amp
rpm	1425
wattage	180 w

**Tachometer**

Model	Toshniwal hand tachometer type 630
Range	30 to 50000 rpm
Accuracy	1.333 between 30 - 150 44.44 between 100 -500 1.333 between 300 -1500 44.44 between 1000 - 5000 1.333 between 300 - 15000 444.44 between 10000 - 50000

**Voltage Stabilizer**

Model	Automatic servo voltage regulator - type LLS/VRT-7/89
Current	AC, 50 cycles, 1 phase
Capacity	2.5 KVA, input: 175 - 250 V, output : (230 +/- 1%)V

**Refractometer**

Model	ABBE - 3L, Baucsh and LOMB, USA
Range of measuring	1.30 - 1.71 $n_D$
Scale	0 - 85% (total dissolved solids)

**Weighing balance**

Model	Saratorius, 2004 MP
Accuracy	0.00001 gm

## **4.2 Experimental Device**

### **4.2.1 Apparatus**

A stainless steel cylindrical stirred ultrafiltration cell was used to study the influence of various factors that affect the flux declining phenomenon. A high pressure reciprocating pump and a back pressure regulator were used to maintain and change pressure. The system is provided with two dampeners to reduce the fluctuations in pressure and maintain it at the set point.

A schematic diagram of the experimental set up is shown in figure 4.1. The specification of the stirred UF cell shown in figure 4.2 are as follows:

Material	SS316
Useful volume	650 cc
Residual volume	3 cc
Membrane diameter	76 mm
Effective filtration diameter	<b>65 mm</b>
Maximum Testing Pressure	1000 kPa
Stirring facility	Mechanical

### **4.2.2 Feed Solution**

Limed cane juice was used as the feed solution

### **4.2.3 Design Of experiments**

Experiments were designed to study the effect of three independent variables viz. concentration, pressure differential and MWCO at constant stirrer speed (600 rpm), on flux and rejection. During an experiment all the three variables were kept constant and flux decline behaviour with time was measured. The concentration of the feed solution ( in RI ) was taken as 10, 13 and 16 brix, respectively and the operating pressures chosen were 476, 680 and 884 kPa, respectively. The three UF membranes used were of 10000, 15000 and 20000 MWCO.

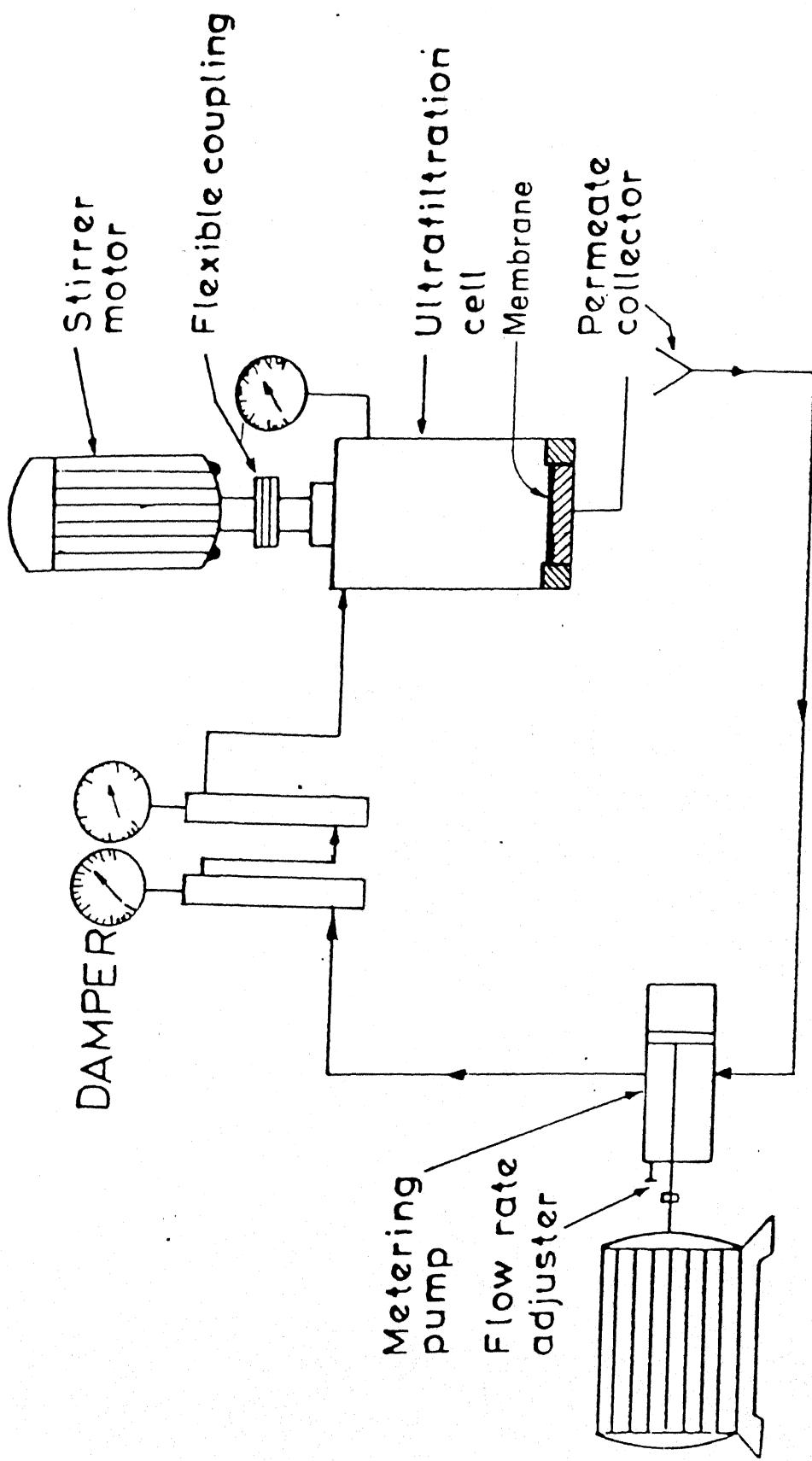
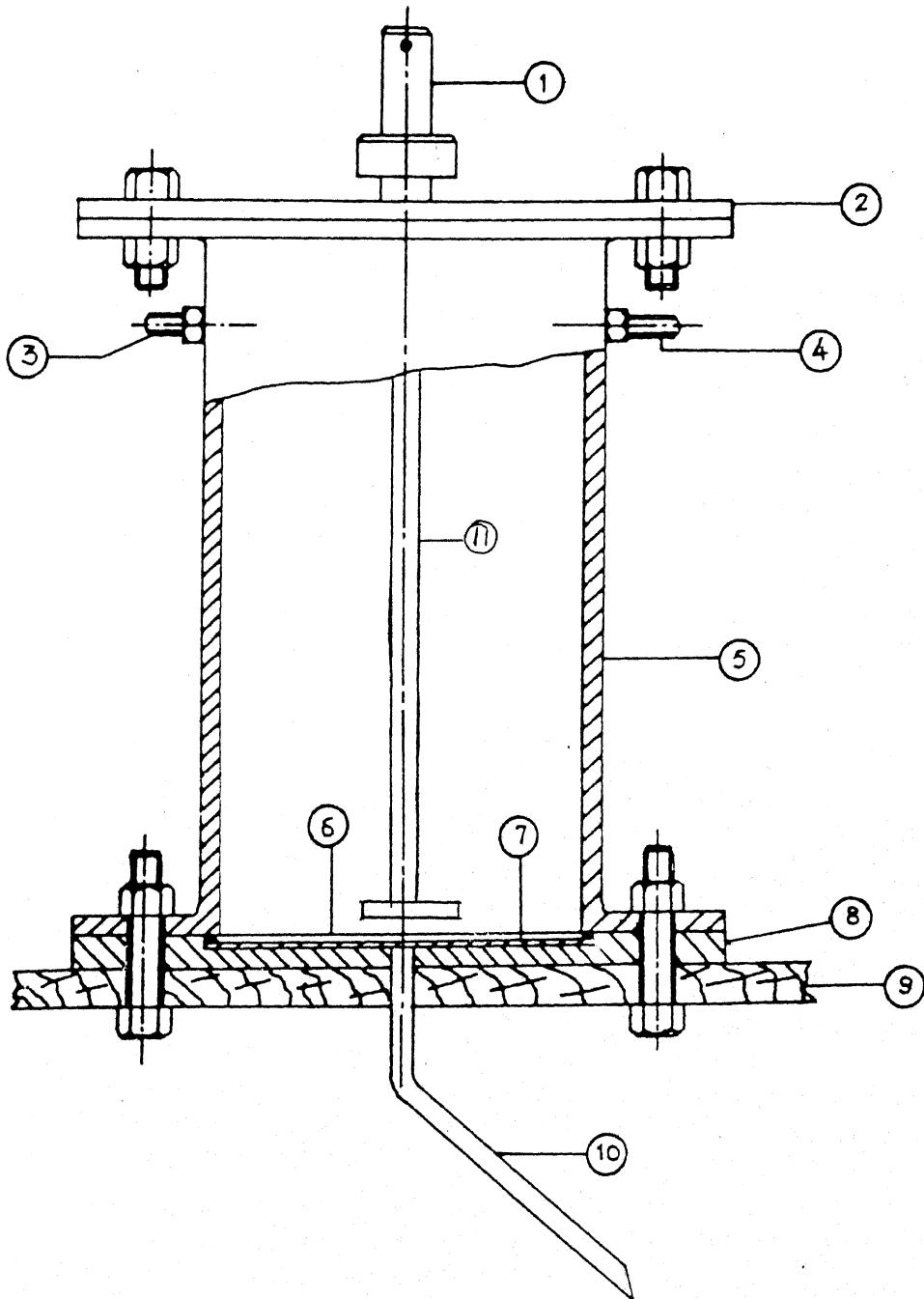


Fig 4.1 A schematic diagram of the experimental set up



**Fig 4.2 Ultrafiltration cell**

- 1.Fitting for stirrer motor.
- 2.Top flange.
- 3.Feed inlet.
4. Pressure line connector.
5. Stainless steel cell.
6. O - Ring.
7. Membrane.
8. Base flange.
9. Fixed support.
10. Permeate collector.
11. stirrer.

## **4.3 Experimental Procedure**

### **4.3.1 Liming Of The Raw Cane Juice.**

The raw cane juice, obtained from the local vendors, was treated with milk of lime to raise its pH from 5.2/5.4 to 8.0. The liming was done at room temperature (22-23°C). It is done in order to settle the suspended solids and produce a clear juice. The treated juice was kept for about two hours to facilitate the settling of the solids. Then, the clear brownish red supernatant limed juice was siphoned out. This supernatant juice is that which is subjected to the UF membranes after prefiltering it, using one of the following methods:

1. Packed bed :It is a 16 cm bed of glass beads( 1mm diameter) and 60 - 120 mesh silica gel . The height of silica gel bed is approximately 8 cm.
- 2 Whatmann - 42 filter paper after filtration through the packed bed, under vaccum.
3. Whatmann-42 filter paper under vaccum.

### **4.3.2 Ultrafiltration Of Limed Cane Juice**

First a membrane is compacted at 1020 kPa(150 psi) for atleast two hours. The compaction pressure should be higher than the highest operating pressure. This procedure is adopted to prevent decline in flux during the experiment due to the compaction of the membrane. During compaction the water flux is continuously measured until it becomes constant. This suggests that membrane has been compacted and is ready for use. Then, water flux at different pressures ( less than 1020 kPa ) are measured to find the hydraulic resistance ( $R_m$ ) of the membrane.

Once the above procedure has been done, the feed solution is then pumped into the cell by the reciprocating pump. The speed of the stirrer is adjusted by regulating the voltage supply with the help of a variate. The rpm of the stirrer speed is measured by tachometer. The pressure level is adjusted with the help of back pressure regulator (BPR). The permeate is collected and recycled back to the feed tank intermittently to keep the concentration of the feed constant.

To measure flux , a 25 cc measuring cylinder was taken. Time after collection of every 5 cc of permeate was noted. The experiments were stopped

after the flux reaches a constant value.

After each run, the cell is thoroughly rinsed with distilled water. The membrane is also washed with distilled water and kept in the same for atleast twenty four hours before using for the next experiment. However, every time the hydraulic resistance of the membrane is found before using it for next experiment. This is done to quantitatively find the change in the permeability of the membrane. This procedure was repeated for each set of operating conditions.

#### **4.3.3 Measurement Of The Concentration**

Refractive index calibration method is used to determine the concentratiton of the raw cane juice and the limed cane juice. It is given in figure 5.1. To find the amount of total dissolved solids in the solution , the following procedure was adopted:

- 1.10 cc of the solution was taken in a petredish that was preweighed.
2. The solution was evaporated in an oven at  $100^{\circ}\text{C}$  and the mass of the remaining solid was weighed to calculate the concentration ( total dissolved solids).
- 3.In the calculations , however, always mesaured RI of the solution was used to estimate the concentration of the solids in the solutions.

#### **4.3.4 Measurement Of The Viscosity And Density**

Viscosity of limed cane juice is determined by Ostwald's viscometer at a constant temperature( 22- 23 $^{\circ}\text{C}$ ). A correlation between viscosity (cP) and concentration (RI) was developed by fitting a polynomial curve(ref. Appendix A). The fitted curve was used in the calaculaion and analysis .

Similarly, the density (g/cc) of limed cane juice is detemined with help of specific gravity bottle (ref. Appendix A) and correlated to the concentration (RI) of the juice.

## **Chapter 5**

# **RESULTS AND DISCUSSIONS**

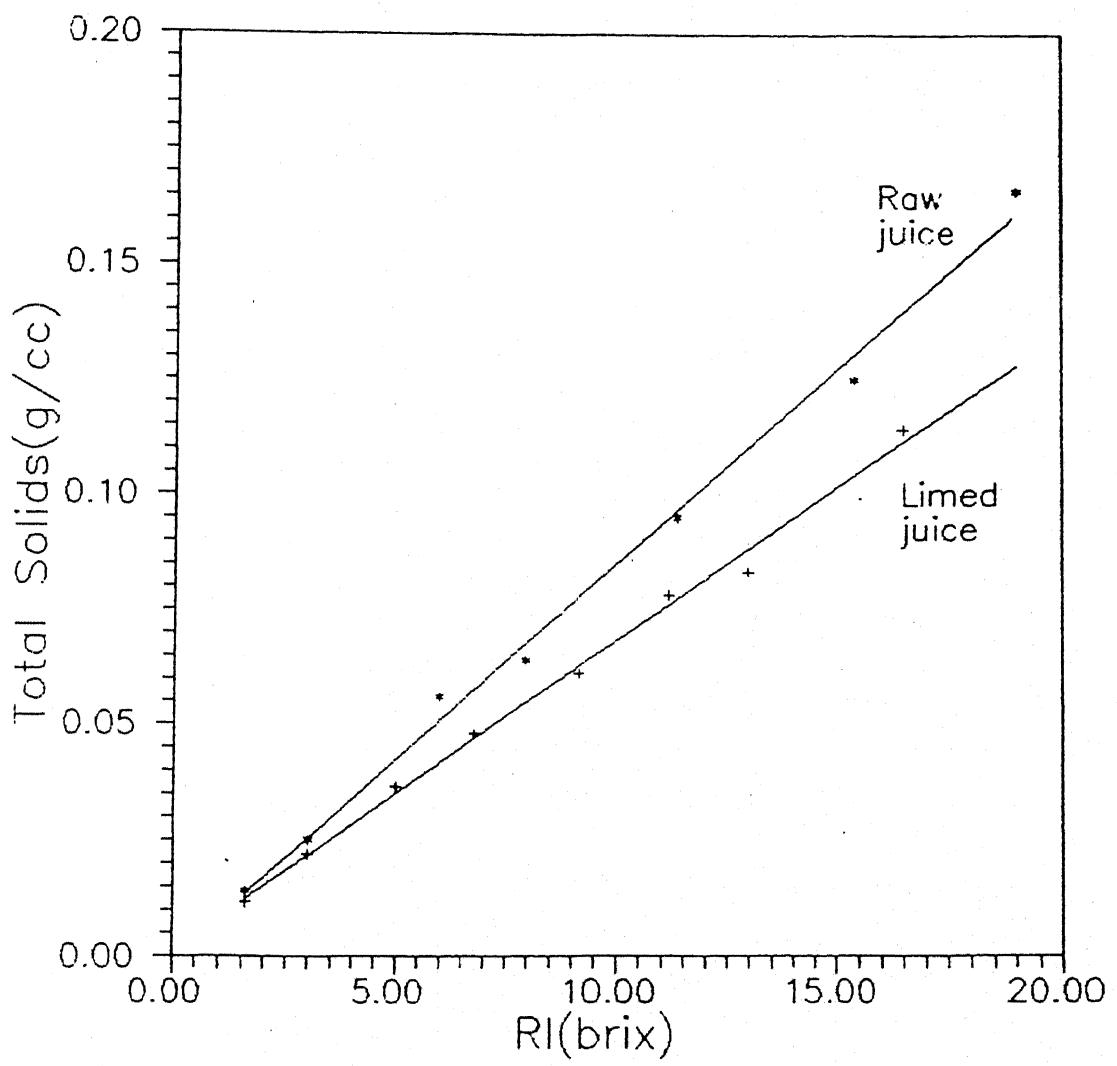
### **5.1 Pretreatment Of Raw Sugarcane Juice**

#### **5.1.1 Cold Liming of the raw juice**

The raw sugarcane juice is treated with the milk of lime to coagulate the suspended solids and produce a comparatively clearer juice. This standard clarification process removes mud, suspended particles, colloidal matter, proteins, waxes etc. This reduces the amount of macromolecular suspended matter and also the load on membranes and its chances of early fouling are reduced. The amount of matter settled or removed due to liming can be found from figure 5.1. It appears from the figure that as the concentration of raw juice increases amount of solids settled increases. However, on calculations it was found that approximately 20% of the total dissolved solids are removed on liming at each concentration of juice. This is obvious as the liming is simply restricted by control of pH only (i.e at 8.0).

#### **5.1.2 Prefiltration Of Limed Juice**

The clarified juice obtained after liming, although is free of mud but still contains high molecular weight proteins, organic colouring matter etc. These dissolved matters may result in decline in flux and cause membrane fouling, when this feed is subjected to UF. Therefore, some pretreatment of clarified juice was felt necessary and some of the methods used were :



**Fig 5.1** Concentration Vs refractive index calibration curve

#### **5.1.2.1 Filtration Through a Silica Gel Bed**

The clarified limed juice was passed through a differential packed bed under vacuum. The bed comprised of glass beads(1 mm diameter) , topped by 8 cm of 60 -120 mesh size of silica gel. The height of the total bed was approximately 16 cm. The colour of the bed becomes yellow after treatment , indicating the absorption of some of the colouring matter. A reduction in refractive index (RI) was also observed. Some typical data have been reported in table 5.1. This reduction in RI retention of high molecular substances and suspended matter on bed.

#### **5.1.2.2 Filtration Through Whatmann -42 Filter Paper After Packed Bed Filtration**

The limed juice after passing through packed bed was passed through filter paper under vacuum not much change in RI was observed as can be seen from typically reported data in table 5.2. It was experienced that the filter paper have to be changed atleast twice to filter two litres of limed juice as it used to get plugged. On weighing the dried filter papers, it was found that they retained about 0.3 gm/l of solids.

#### **5.1.2.3 Filtration of Limed Juice Directly Through Filter Paper**

On filtering the clarified lime juice directly through Whatmann-42 under vacuum , a greenish brown layer formed on the filter paper that plugged the pores,making it compulsory to change the filter paper 3-4 times for filtering two litres of juice. On measuring it was found that 0.5-0.6 gm total solids per litre of juice were retained.

## **5.2 UF Of Pretreated Cane Juice**

### **5.2.1 Estimation Of Sucrose Rejection**

The aim of the work is to recover sucrose and remove all other substances from the pretreated limed juice. Hence, it was necessary to estimate the individual concentration of the sucrose, both in feed and permeate. However,only the bulk concentration of the feed and permeate both were measured in RI.This method was adopted as the analysis of the juice was difficult.Further, the composition varied with place , crop and season.

Concentration	
Limed juice RI (brix)	Filtered Limed juice RI (brix)
16.3	15.4
17.7	16.8
18.7	17.7
19.7	18.6
22.0	20.0

Table 5.1 Refractive index of limed juice before and after filtration through packed bed.

Concentration	
Limed juice RI (brix)	Filtered juice RI (brix)
8.7	8.4
9.6	9.3
10.3	10.1

Table 5.2 Refractive index of limed juice before and after filtration packed bed and filter paper.

Hence, the calculations were based on the analysis available from the literature (table 2.1). Further, no data was available on the composition of limed juice. Hence, it had to be generated based on certain assumption (considering 20% solids settled during liming) described in chapter 3. Table 5.3 gives the theoretically developed composition of the limed juice. In the table one can note that the constituents are divided in two distinct groups - permeable solutes and nonpermeable solutes, which is done with respect to the UF membranes (10000, 15000, 20000) used in the present work.

As it has been mentioned earlier that the concentration can be estimated only by measuring RI which for a mixture gives the combined RI of most of its constituents. RI is an extensive property (additive in nature). This was verified in the present case (ref. Appendix B). Therefore, in order to estimate RI of an individual solute (say sucrose), the RI of the mixture is multiplied by its weight fraction. Therefore, in order to estimate the concentration of sucrose in feed and permeate, the RI of the solution is multiplied by the weight fraction of sucrose in the respective solution (0.714 in feed and 0.83 in permeate).

### **5.2.2 Influence Of Different Parameters On Flux And Rejection.**

#### **5.2.2.1 Effect Of Pressure On Flux.**

The variation of flux against pressure have been plotted for three different membranes (10000, 15000, 20000) as a function of limed juice bulk concentration (10, 13, 16 brix) as shown in figures 5.2-5.4. As expected the flux increases with increase in pressure. In case of polyamide 15000 membrane (fig. 5.3) the rate is much more than the cellulose acetate membranes viz. 10000 and 20000 (figs. 5.2, 5.4). All the fluxes show similar trend of decline with time at different bulk concentration of feed solution.

The motion of the molecules through 20000 membrane is essentially due to the convective flow governed by poiseuelle law while as the MWCO size decreases, the flow of molecules due to diffusion becomes more and more predominant. Therfore, the attainment of plateau (where pressure has negligible effect on flux) for 10000 and 15000 membrane is beyond 884 kPa.

Constituents	Feed		Permeate Dry Basis(%)	
	Basis(%)			
	Wet	Dry		
Water	75.77	-	-	
Total solids	24.33	100	85.69	
Permeable solutes	20.85	85.67	100	
- Ash	0.32	1.32	6.33	
- Sugar	17.37	71.37	83.30	
- Pentosans	3.16	12.99	15.16	
Non permeable solutes	3.48	14.30	-	
- Lignin (fibre)	2.53	10.40	-	
- Nitrogenous bodies	0.49	2.01	-	
- Fats and waxes	0.14	0.58	-	
- Pectins and gums	0.32	1.32	-	

Table 5.3 Generated composition of limed juice.

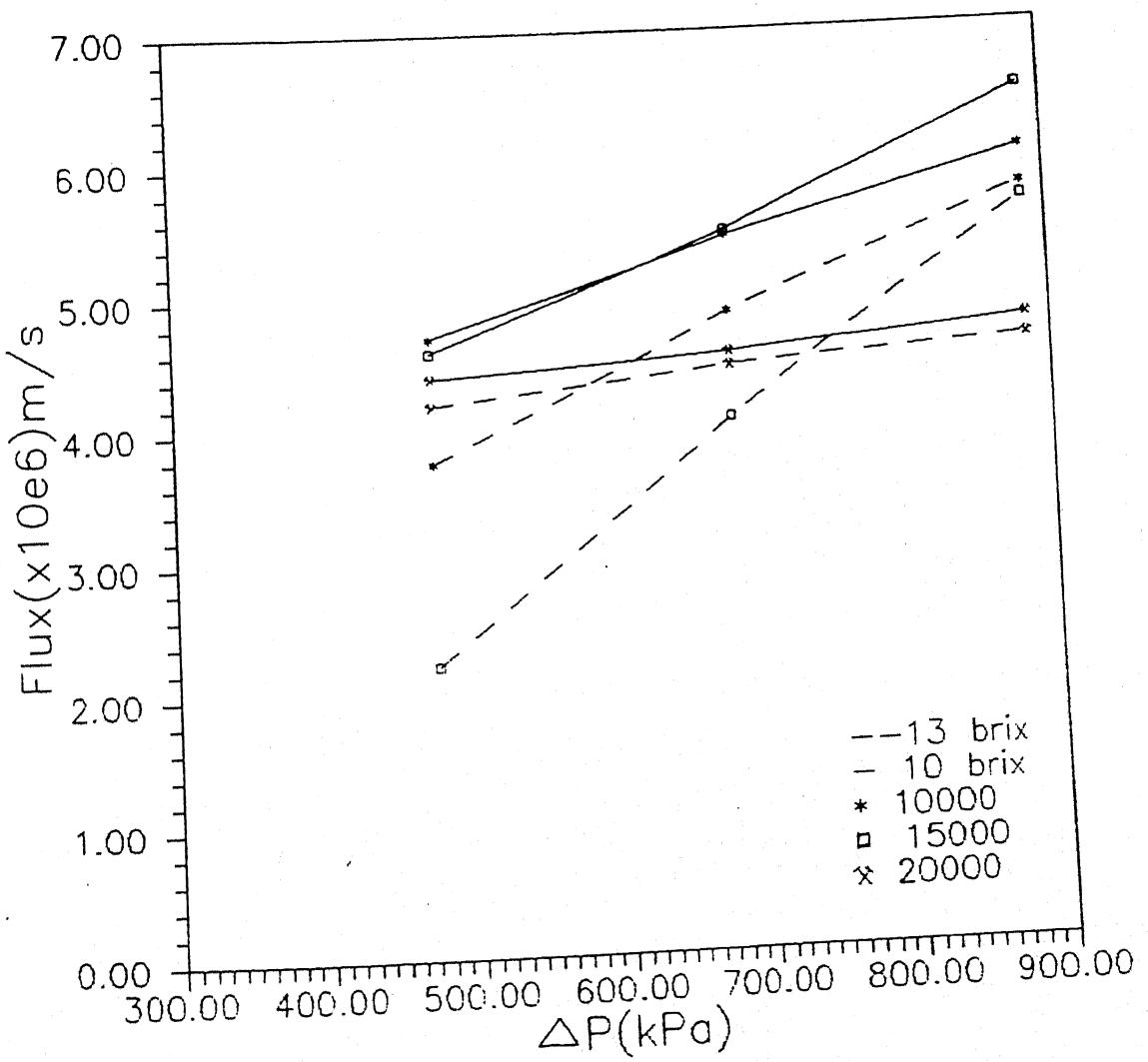


Fig 5.5 The comparative effect of membranes on flux as a function of pressure

Figure 5.5 shows that the flux through 10000 and 15000 membranes is higher than 20000 membrane. This is because 15000 membrane is a diffuse cut-off membrane (large pore size distribution) whereas 10000 and 20000 membranes are sharp cut-off membranes. In a diffuse cut-off membrane, therefore, even if there are few pores of large diameters (greater than 15000), they affect the flux considerably as flux is directly proportional to the square of pore diameter. The second reason is that the hydraulic resistance of the membrane ( $R_m$ ) of 20000 ( $7.68 \times 10^{13} \text{ m}^{-1}$ ) is much higher than 10000 ( $3.96 \times 10^{13} \text{ m}^{-1}$ ) and 15000 ( $3.13 \times 10^{13} \text{ m}^{-1}$ ) membranes.

#### 5.2.2.2 Effect of Bulk Concentration on Flux

It is a known fact that with the increase in the bulk concentration of feed, the permeate flux decreases. This is what is verified by the figures 5.6 - 5.8, where flux is plotted against bulk concentration as a function of pressure. This is due to increase in osmotic pressure which reduces the effective pressure gradient.

In figures 5.6 and 5.7, one can observe that the decay in flux is rapid at 884 kPa compared to that at low pressures (476 and 680 kPa), where decay is gradual. This may be due to the reason that at high pressure the bulk diffusion of macromolecules is slow which thereby contribute in the rapid growth of the layer adjacent to the membrane causing a sharp decline in flux.

In figure 5.8 one can see that the behavior of 20000 membrane is somewhat different, where at low pressures decline is slow. Further, it may be seen that at much higher concentrations the decline might follow a sharp trend. This is because 20000 membrane being a large pore size membrane tends to pass most of the low molecular weight substances and retain large macromolecules (like proteins, dextrans, etc.), which are likely to form gel like layer, but offer a negligible osmotic pressure. Thus the pressure difference across remains unchanged. While, as the pore size of the membrane decreases (e.g. 10000 and 15000) the flow due to diffusion becomes more and more predominant compared to convection. The increase in concentration near surface also increases the viscosity (ref. Appendix A) which further decreases the flux as flux is inversely proportional to viscosity.

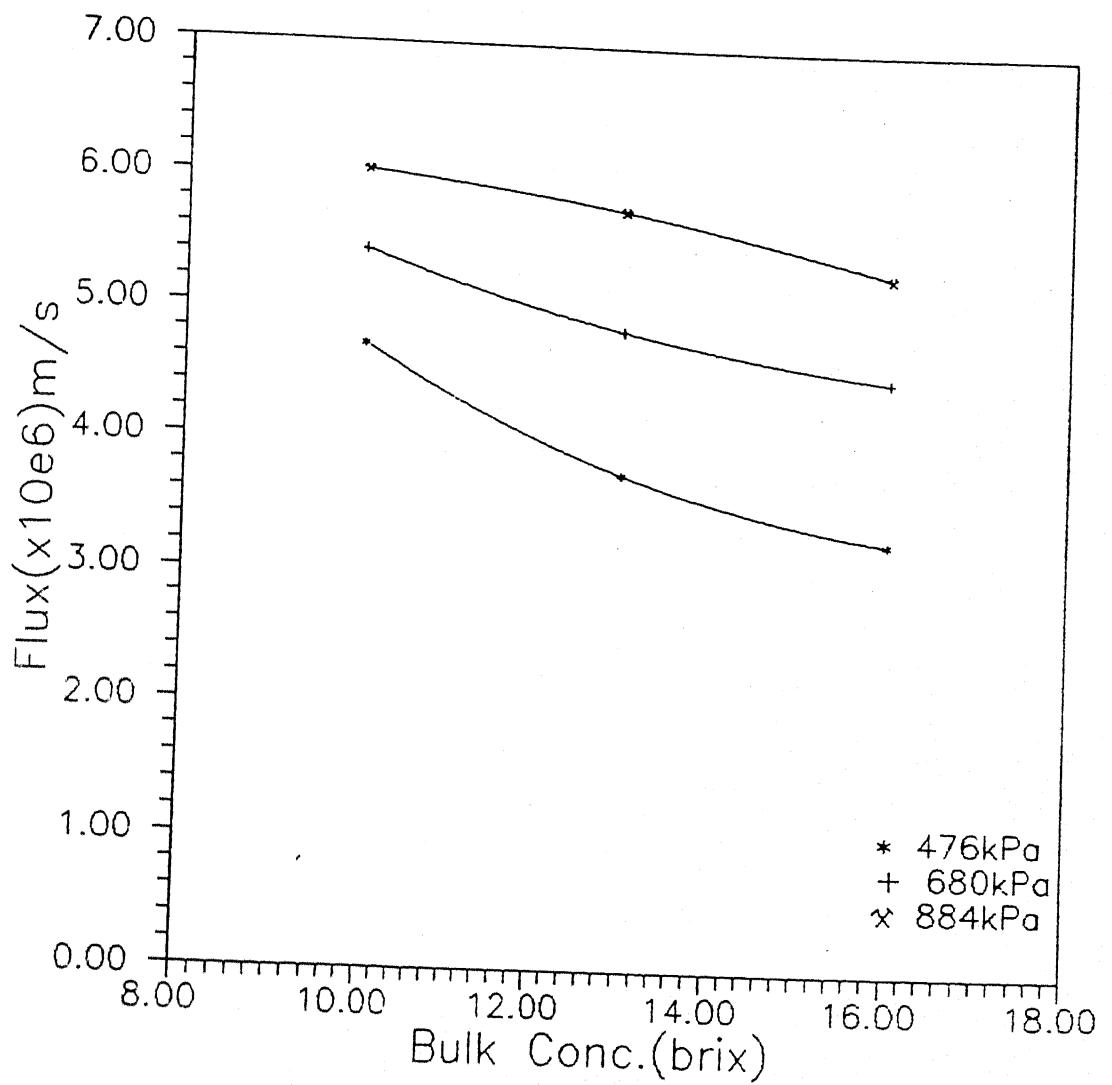


Fig 5.6 Effect of bulk concentration on permeate flux for 10000 membrane

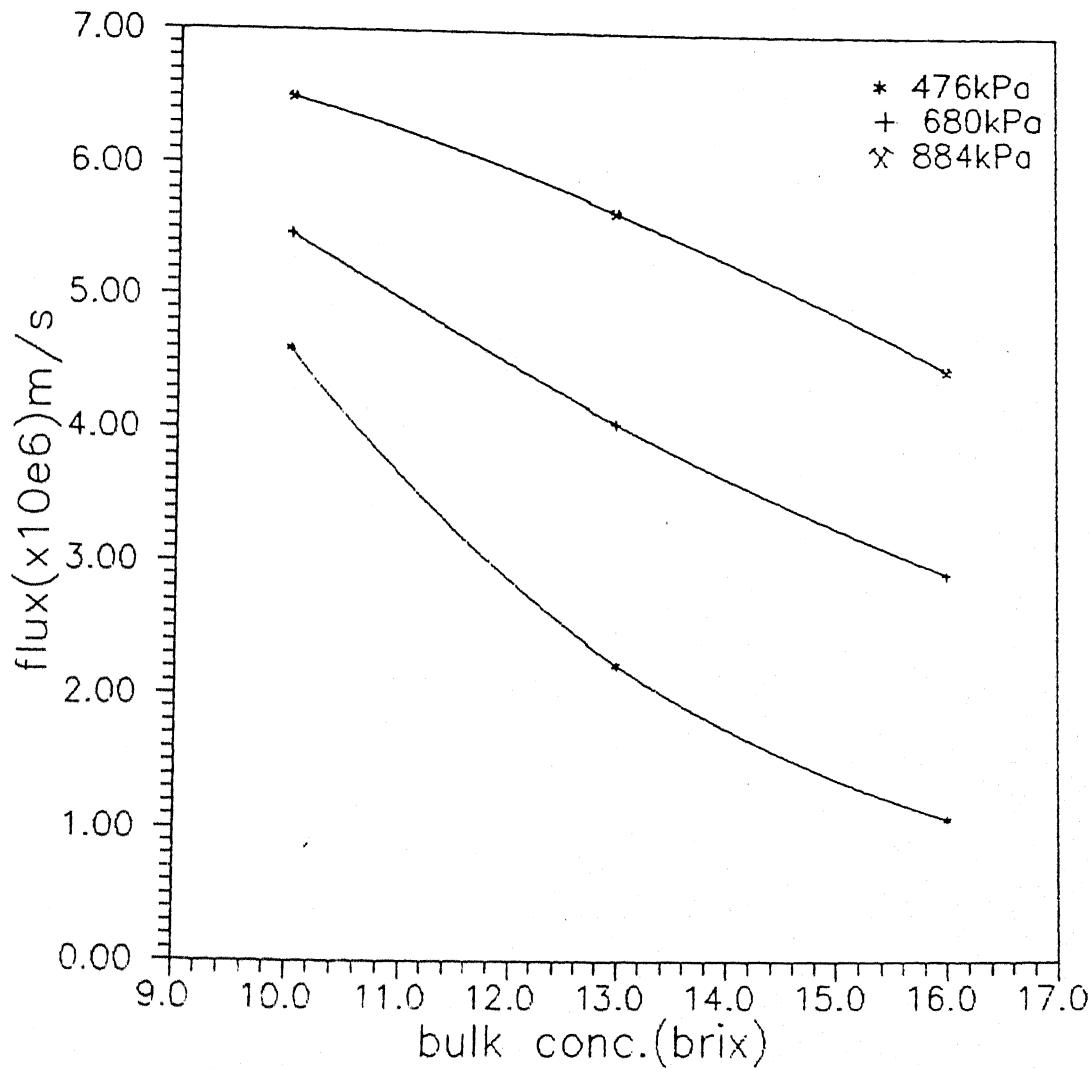


Fig 5.7 Effect of bulk concentration on permeate for 15000 membrane

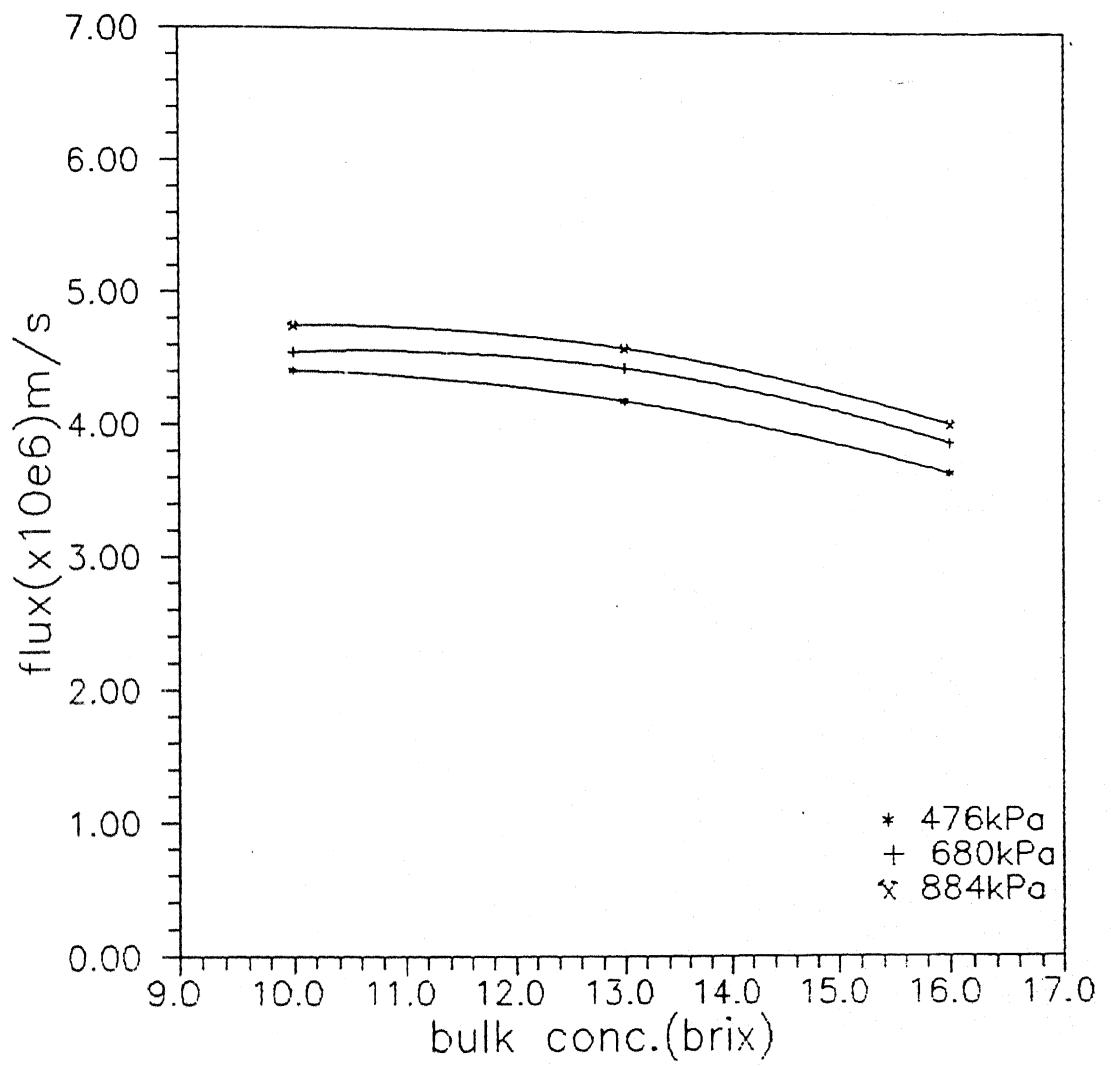


Fig 5.8 Effect of bulk concentration on permeate for 20000 membrane

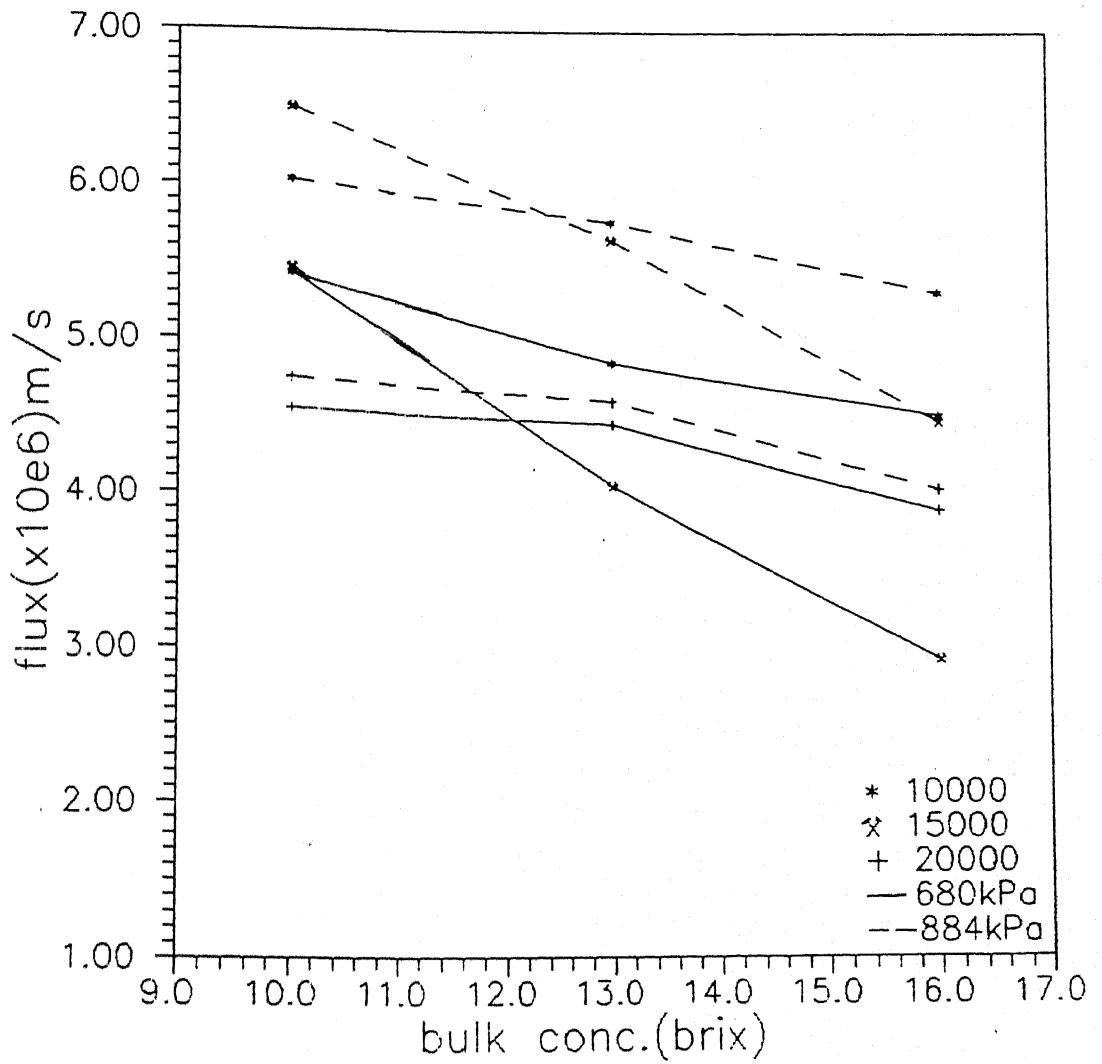


Fig 5.9 The comparative effect of membrane on flux as a function of bulk concentration

### 5.2.2.3 Effect of Pressure on Rejection

The variation of total solid rejection and sucrose rejection with change in pressure at a constant bulk concentration for each membrane is shown in figures 5.10 and 5.11. In most cases the rejection decreases with increase in pressure. However, in the present case the rejection follows an opposite trend. One possible reason for this may be that cane juice forms a dynamic membrane [18], the effect of which is significant at high pressures. This is because at high pressure there is an increase in convective flux towards membrane which enhances the growth of the dynamic layer. This dynamic membrane formed mainly by larger molecules, restrict the passage of the low molecular weight (LMW) substances (sucrose), thereby decreases the permeate concentration (and hence increases the rejection of sucrose).

It has been shown in figure 5.10 and 5.11 that sucrose rejection was considerably less than what was calculated by total brix of the solution. This is notable finding as it shows that higher cut-off membranes may be used for removing macromolecular impurities (other than sucrose) and premeating more or less the sucrose content of the juice.

Further, the permeate was obtained as a transparent and clear solution, except that it was slight yellowish. The yellow colour darkened with increase in concentration. For 10000 membrane at 476 kPa, it was almost colourless whereas for 15000 membrane it was pale yellow. It may be pointed out here that total rejection, infact, does not signify much as it was measures on the basis of bulk concentration of the mixtures of permeate and feed.

One distinct observation that can be made from the figures is that for a 10000 membrane the rejection increases sharply at pressures greater than 680 kPa while for 15000 and 20000 membranes the increase is almost linear from 476 to 884 kPa, this may be because 10000 membrane is a smaller pore size membrane where transport is largely due to diffusion (hence slower rate). Secondly, with increase in pressure the rate of growth of dynamic layer increases rapidly. This further restricts the passage of sucrose through such compact layer.

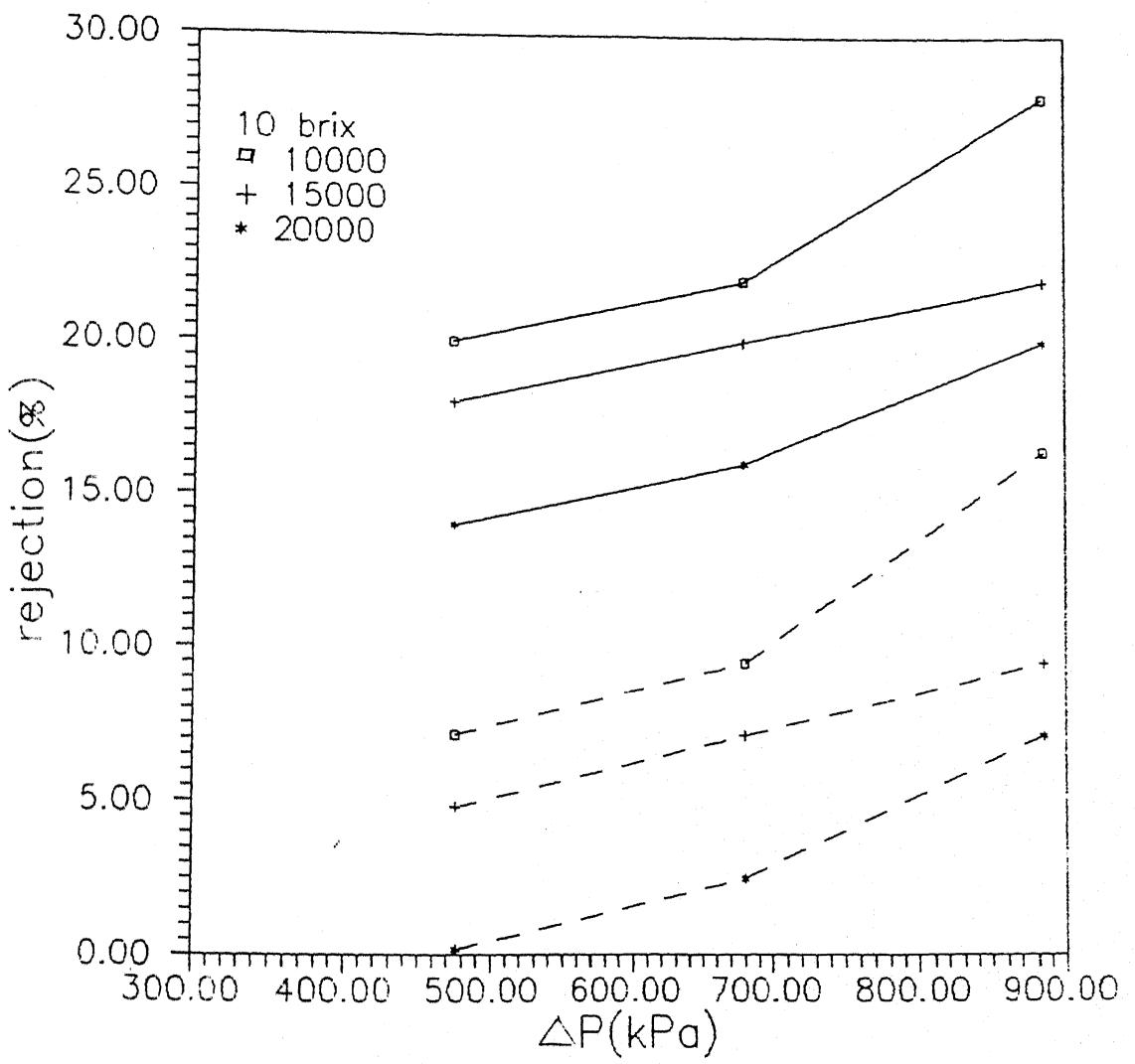


Fig 5.10 Comparison of total rejection and sucrose rejection

#### **5.2.2.4 Effect of Bulk Concentration on Rejection**

In figure 5.12, percent sucrose rejection is plotted against bulk concentration (brix) for different membranes as a function of pressure. It can be observed that as the bulk concentration increases, the rejection also increases. However, rate of increase is high upto 13 brix while after it the increase in rejection is gradual. The increase in bulk concentration increases the boundary layer surface concentration. This enhances the growth of dynamic layer and hence, rejection increases as was explained earlier. It appears beyond 13 brix bulk concentration, there is not much increase in surface concentration implying that the surface concentration reaches a critical value beyond which it may be forming gel. Furthur, gel concentration by its nature remains constant hence passage of solute through gel does not get restricted, thus, keeping the increase in rejection gradual. However, increase in thickness of dynamic layer reduces the flux as is already evidenced from figures 5.6 and 5.7. An obvious observation from the figures is that as the MWCO increases the rejection decreases.

### **5.3 Prediction Of Limiting Flux**

One of the main reasons for the slow acceptance of ultrafiltration treatment for clarifying and concentrating liquid solutions and suspensions is the decline of flux with time thus, adversely affecting the output rate. This decline of flux is due to concentration polarization. The consequence of this concentration polarization which ultimately leads to fouling of membrane, have several other implications i.e. formation of polarized layer, adsorption on the membrane surface, plugging and clogging of pores etc. In general these all contribute to an extra resistance. It is also a well known fact that concentration polarization cannot be avoided but can be minimized by optimizing the operating conditions and proper case study of the feed on the membrane. In the present work the limed juice subjected to ultrafiltration did exhibit drop in flux with time and some deposition on the membrane surface was also observed. Thus, it became necessary to study the limiting flux phenomenon during UF to predict and analyse the flux decline. In the present work, two established models [27,28,31-33] were chosen to steady this aspect. Furthur, the analysis through such models would enable us assess one or some better conditions to ultrafilter limed cane juice. The chosen models are :

1. Osmotic pressure polarized layer model [21,31,32 ]
2. Cake filtration model [28,30]

### 5.3.1 Osmotic Pressure Polarized Model

#### 5.3.1.1 Estimation and Influence of Polarized Layer Resistance on Operating Conditions

To estimate the polarized layer resistance an iterative technique was adopted [ch 3]. The algorithm for which is given in Appendix C. The algorithm was repeated for each experimental run and polarized layer resistance ( $R_p$ ) as well flux value was calculated at each time interval. Figure 5.13 shows the calculated flux for a particular bulk concentration and pressure differential. Since, the osmotic pressure model described in eqn. 3.1 does not account for any extra resistance that builds up as a function of time, hence, the calculated flux is constant for a run.

The figure 5.13 shows that deviation between  $J_{cal}$  and  $J_{expt}$  increases with time. Furthur in figure 5.14 and 5.15, polarized layer resistance increases with respect to time, which has been extimated from eqn.3.13. The influence of polarized layer resistance on operating conditions can be seen in figures 5.16 and 5.17. Figures 5.16 and 5.17 clearly shows that  $R_{ps}$  ( $R_p$  calculated at steady state) increases with increase in concentration and pressure. It is clear from the figures that in lower pore size membrane the bulid up polarized layer is more than in case of large pore size membrane. Also as the bulk concentration increases, polarized layer resistance also increases. This is more evident in figure 5.11 where beyond 13 brix rate of increase of  $R_p$  with bulk concentration is much higher. Furthur, it depicts that 15000 membrane offers highest  $R_p$ . This may be attributed to the fact that 15000 membrane is a PA membrane of wide pore size distribution where as 10000 and 20000 membranes are CA membranes of narrow pore size distribution.

### 5.3.2 Cake Filtration Method

To predict the specific cake resistance in a stirred UF cell, another model proposed [30] was used. In this the model has been described in

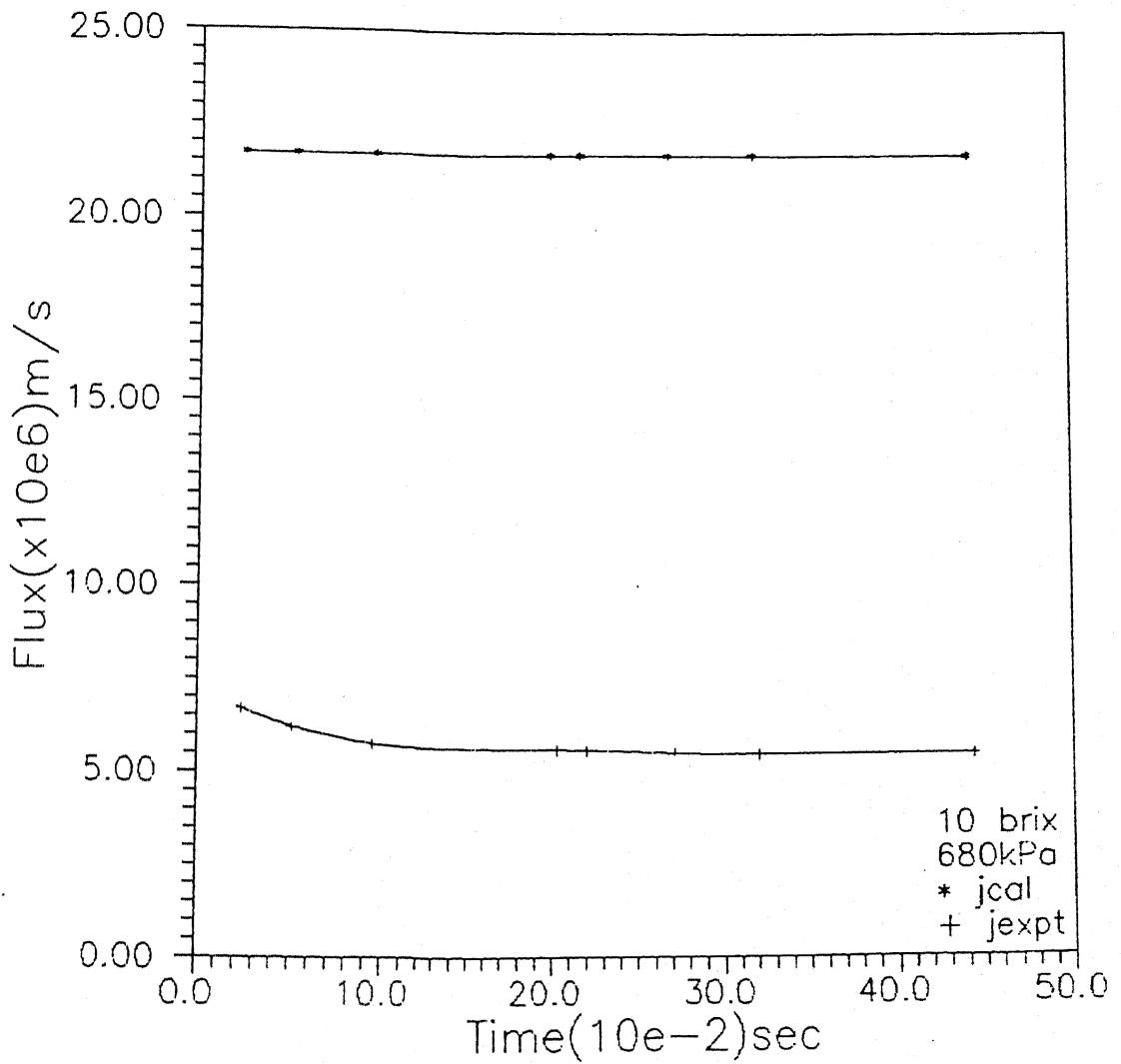


Fig 5.13 Comparison of calculated and predicted flux for 15000 MWCO

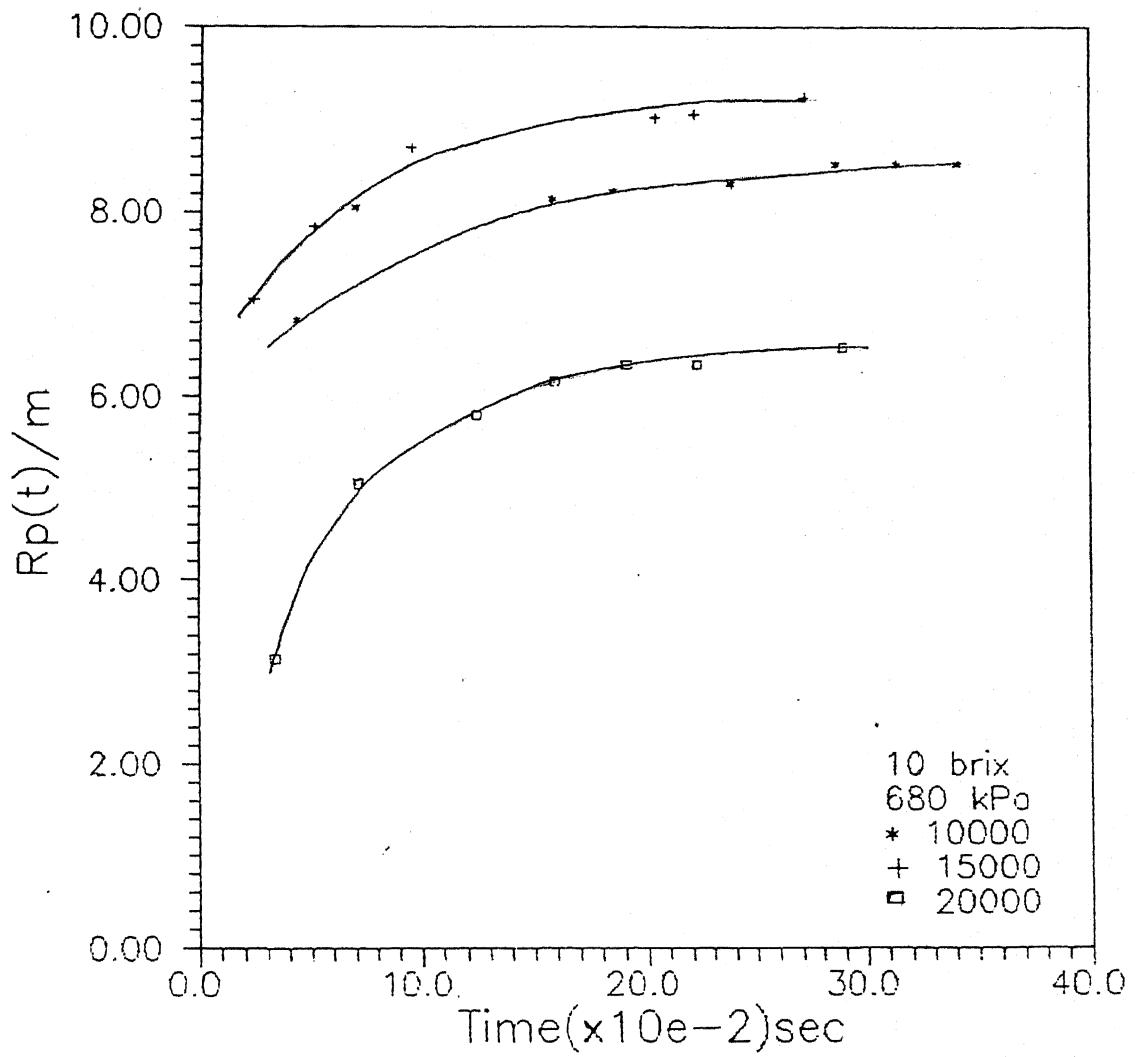
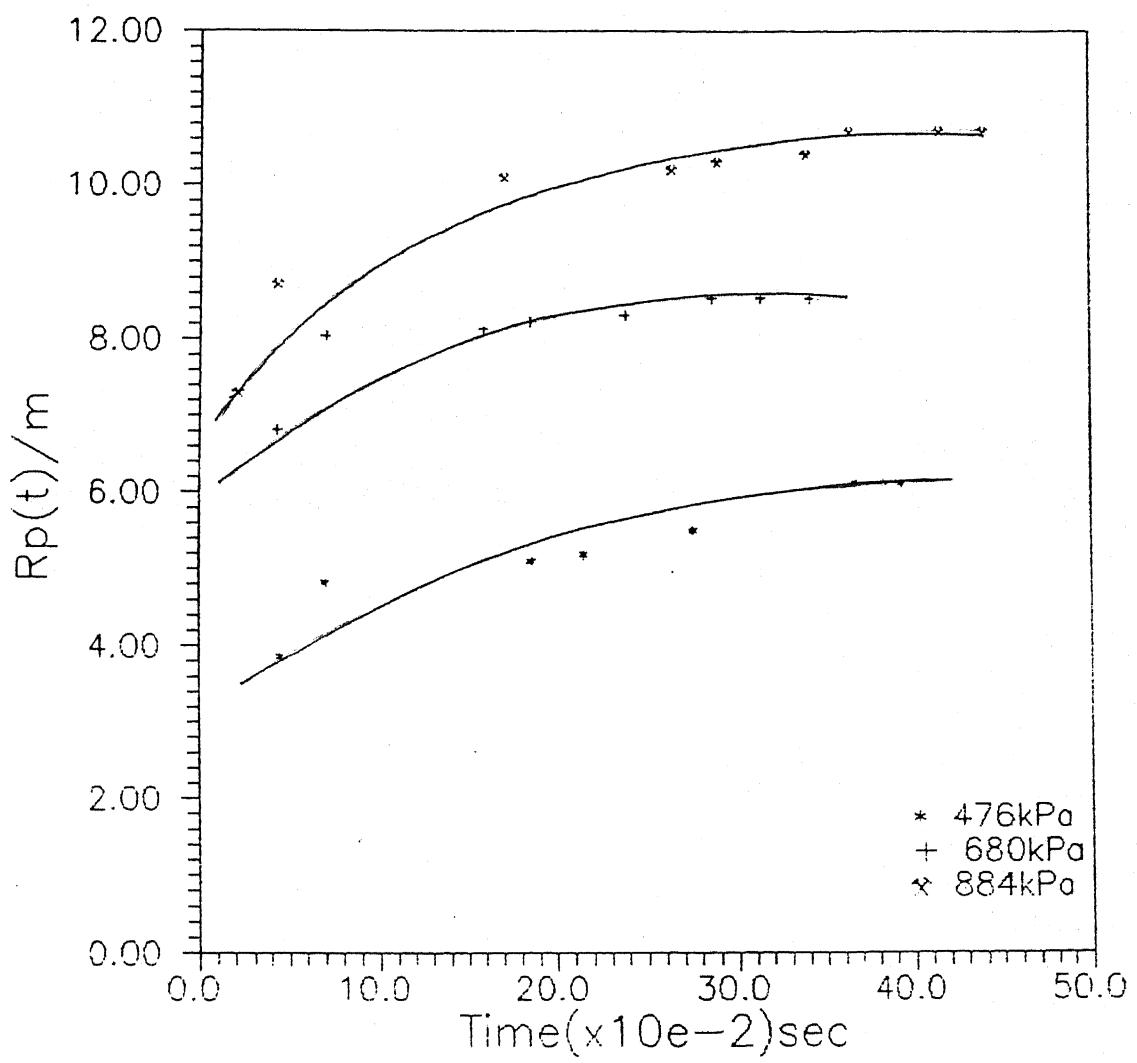


Fig 5.14 Variation of polarised layer resistance with time for different mem branes



**Fig 5.15** Variation of polarised layer resistance as a function of pressure with time for 10000 membrane for 15 brix

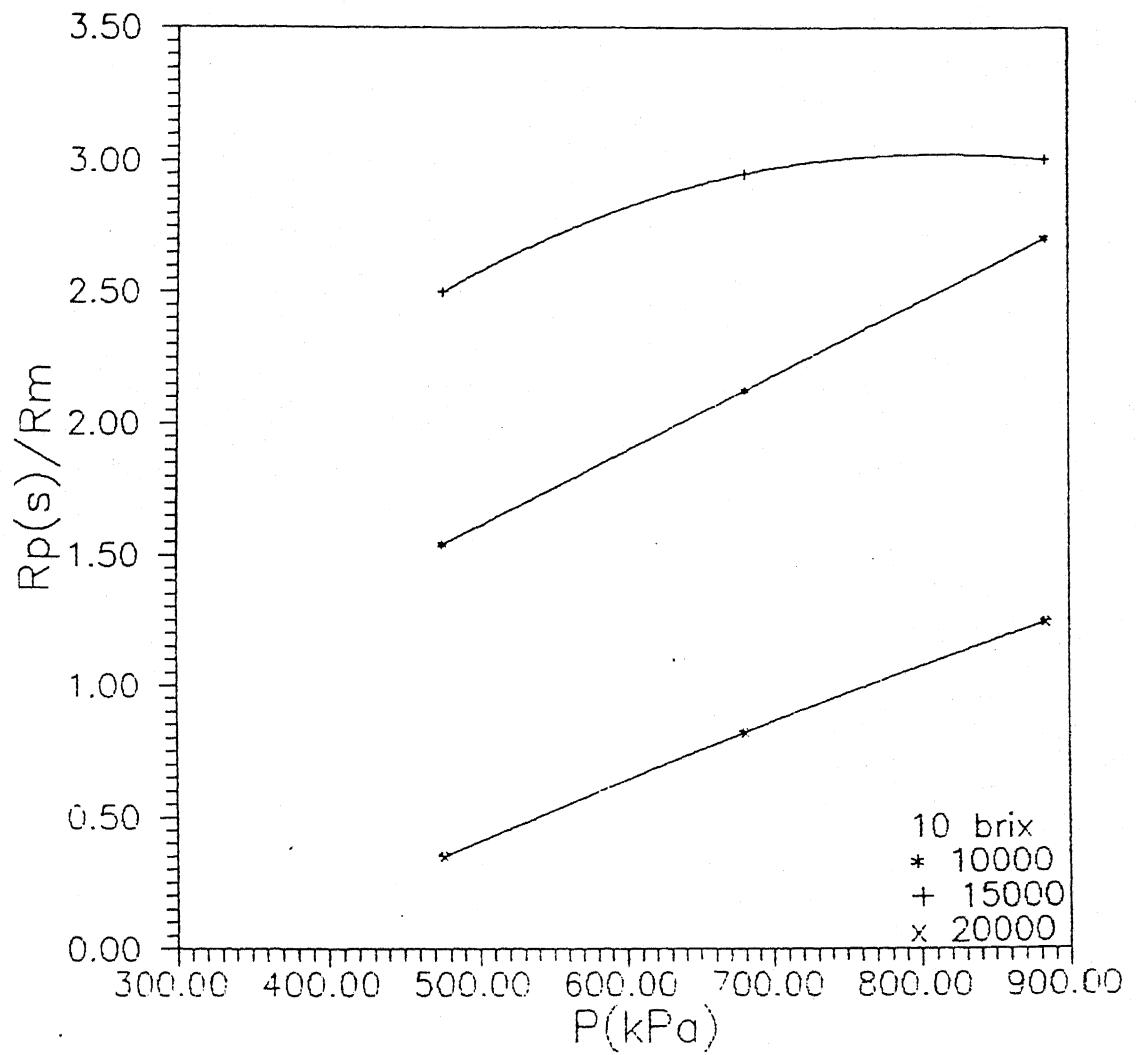


Fig 5.16 Effect of pressure on polarised layer resistance

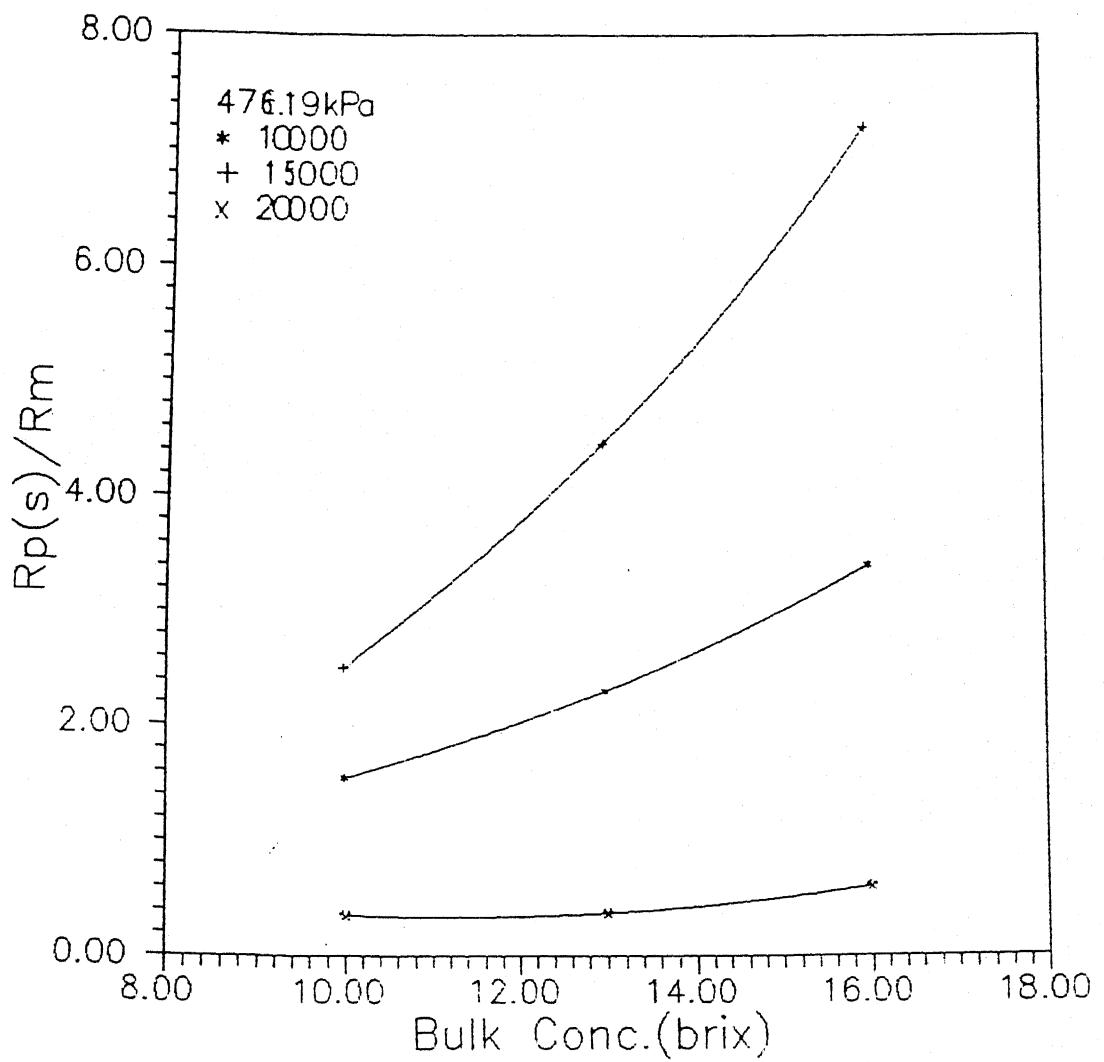


Fig 5.17 Effect of bulk concentration on polarised layer resistance

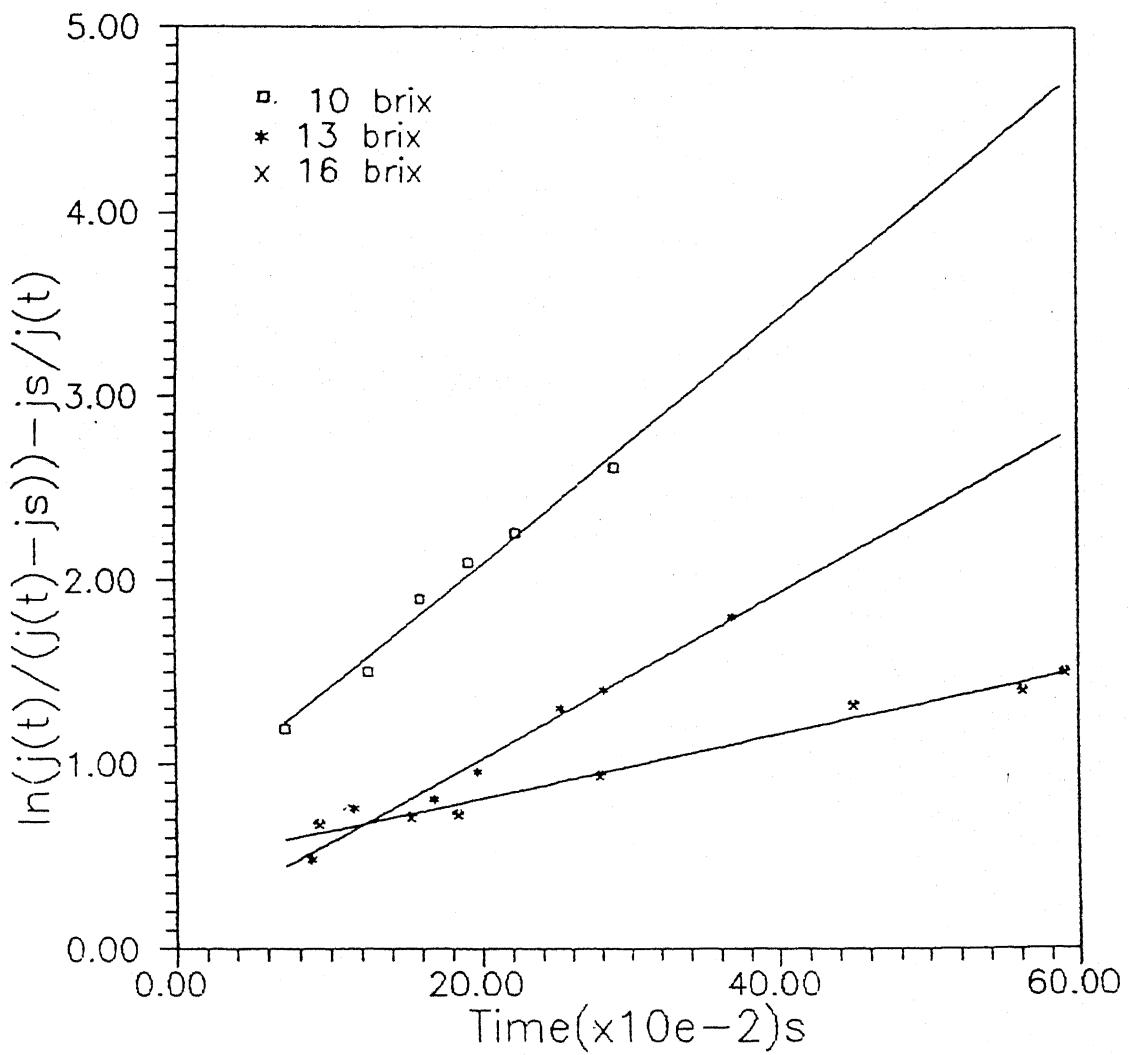
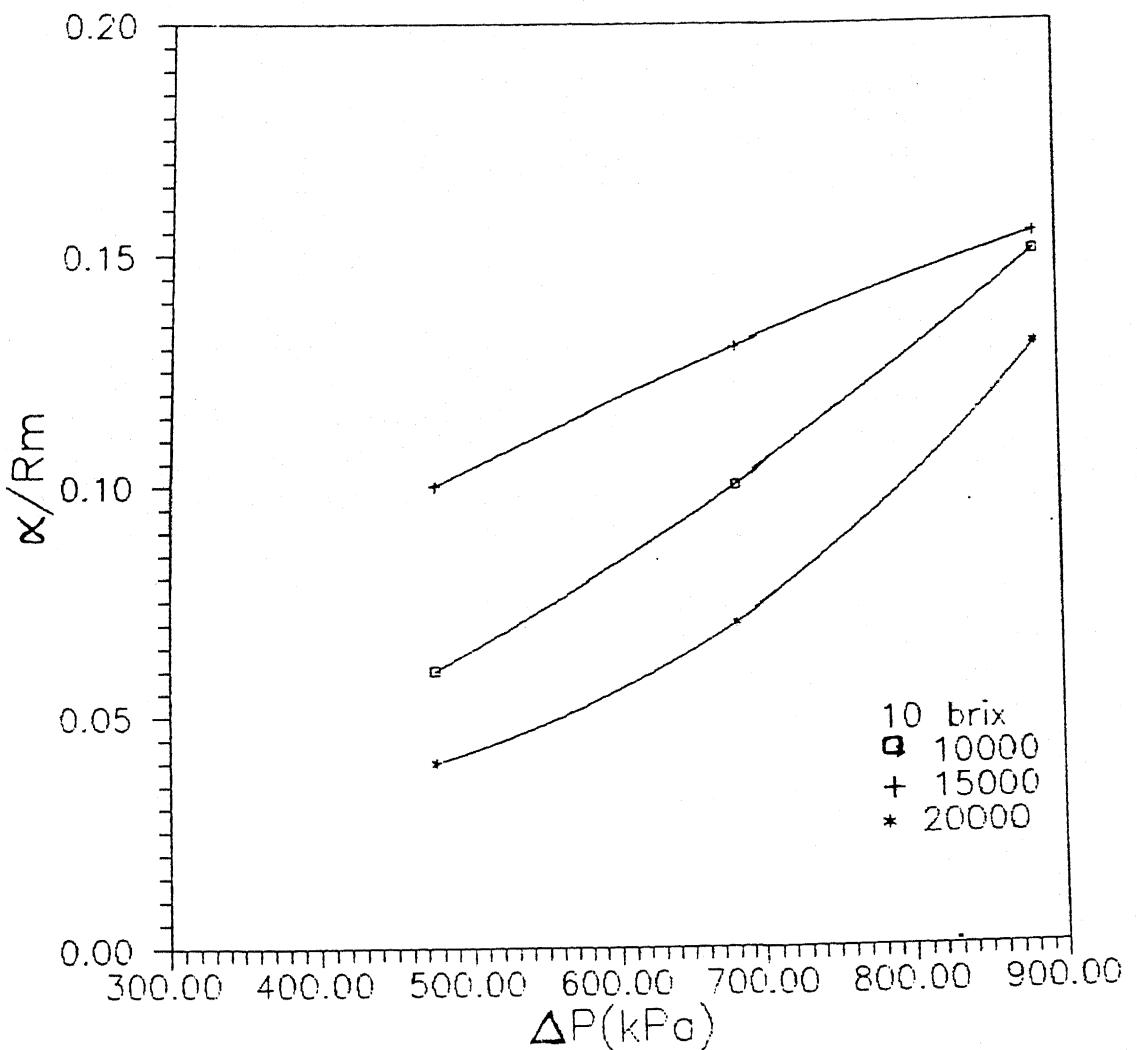


Fig 5.18 Specific resistance of cake for a 20000 membrane



**Fig 5.20** Effect of pressure on the ratio of specific resistance and hydraulic resistance

P = 476 kPa			
	RI = 10 brix	RI = 13 brix	RI = 16 brix
$J_s$	4.71	3.79	3.28
$R_s$	7.10	1.78	11.5
$R_p$	$6.11 \times 10^{13}$	$8.70 \times 10^{13}$	$1.18 \times 10^{13}$
$\alpha$	$2.35 \times 10^{12}$	$1.46 \times 10^{12}$	$1.18 \times 10^{12}$
P = 680 kPa			
	RI = 10 brix	RI = 13 brix	RI = 16 brix
$J_s$	5.43	4.85	4.51
$R_s$	9.5	14.3	17.3
$R_p$	$8.52 \times 10^{13}$	$9.62 \times 10^{13}$	$11.1 \times 10^{13}$
$\alpha$	$4.05 \times 10^{12}$	$4.68 \times 10^{12}$	$2.65 \times 10^{12}$
P = 884 kPa			
	RI = 10 brix	RI = 13 brix	RI = 16 brix
$J_s$	6.03	5.97	5.31
$R_s$	16.4	26.8	27.4
$R_p$	$10.7 \times 10^{13}$	$10.8 \times 10^{13}$	$9.69 \times 10^{13}$
$\alpha$	$6.06 \times 10^{12}$	$12.1 \times 10^{12}$	$1.13 \times 10^{12}$

Table 5.4 Tabulated results of 10000 MWCO membrane

476 kPa			
	10 brix	13 brix	16 brix
$J_s$	4.6	2.23	1.08
$R_s$	4.8	-	7.13
$R_p$	$7.29 \times 10^{13}$	$18.1 \times 10^{13}$	$31.0 \times 10^{13}$
$\alpha$	$2.9 \times 10^{12}$	$1.3 \times 10^{12}$	$1.1 \times 10^{12}$
680 kPa			
	10 brix	13 brix	16 brix
$J_s$	5.47	3.39	2.93
$R_s$	7.1	10.6	12.9
$R_p$	$9.24 \times 10^{13}$	$14.9 \times 10^{13}$	$20.1 \times 10^{13}$
$\alpha$	$4.1 \times 10^{12}$	$12.5 \times 10^{13}$	$4.5 \times 10^{13}$
884 kPa.			
	10 brix	13 brix	16 brix
$J_s$	6.5	3.65	4.47
$R_s$	9.5	14.3	15.8
$R_p$	$9.44 \times 10^{13}$	$11.7 \times 10^{13}$	$15.9 \times 10^{13}$
$\alpha$	$5.7 \times 10^{12}$	$7.6 \times 10^{12}$	$5.8 \times 10^{12}$

Table 5.5 Tabulated results of a 15000 MWCO membrane

476 kPa			
	10 brix	13 brix	16 brix
$J_s$	4.41	4.2	3.66
$R_s$	0.2	-	1.328
$R_p$	$2.82 \times 10^{13}$	$3.0 \times 10^{13}$	$4.65 \times 10^{13}$
$\alpha$	$3.5 \times 10^{12}$	$7.5 \times 10^{12}$	$9.9 \times 10^{12}$
680 kPa			
$J_s$	4.55	4.45	3.89
$R_s$	2.5	3.4	5.67
$R_p$	$6.53 \times 10^{13}$	$7.45 \times 10^{13}$	$0.93 \times 10^{13}$
$\alpha$	$5.6 \times 10^{12}$	$5.6 \times 10^{12}$	$3.4 \times 10^{12}$
884 kPa			
$J_s$	4.75	4.6	4.03
$R_s$	7.1	10.7	11.5
$R_p$	$9.24 \times 10^{13}$	$10.7 \times 10^{13}$	$8.13 \times 10^{13}$
$\alpha$	$9.4 \times 10^{12}$	$6.5 \times 10^{12}$	$8.8 \times 10^{12}$

Table 5.6 Tabulated results of a 20000 MWCO membrane

resistance on different cut-off membranes viz 10000, 15000, 20000. All the results are tabulated in tables 5.4, 5.5 and 5.6. It was thought to analyse the scheme of UF for the recovery of sucrose from the limed cane juice with the help of above stated results. It is obvious that the choice for better scheme would depend upon high flux, low rejection of sucrose and low resistances against flow. However, direct comparision of all the schemes with the results was not possible as the results differed in units and pattern, particularly because of the membranes of varied *characteristics*.

From figures 5.2 - 5.4 one can observe that except in a 15000 membrane, all other membranes do not show any advantage of operating the experiment at high pressure i.e. 884 kpa (130 psi), as the increase in flux is not significant. While in case of 15000 membrane, the flux increases proportionally with the pressure. From the same figures one can see that the flux is highest at 10 brix concentration of the feed. However, it is again the 15000 membrane which gives the highest flux for almost all pressures at 10 brix feed concentration. From figure 5.9, it can be inferred that though the 15000 membrane at 10 brix and all pressures give highest flux but it is 10000 membrane which gives the highest flux at 16 brix concentration of the feed, compared to other membranes.

In figure 5.10 one can notice that 20000 membrane gives the lowest rejection of sucrose at all conditions of pressures and bulk concentration. Hence, it may be the most favourable for industrial applications in terms of recovery of sucrose. However, the flux of 20000 membrane being the lowest, compels one to look for another membrane. The 15000 membrane can be thought as one of such as it also gives quite low rejection of sucrose at low pressures of 476 kPa and 680 kPa (sucrose rejection being 4.8% and 7.17% respectively). But at 476 kPa the flux is considerably low and hence 680 kPa may be chosen as a better operating pressure.

Besides rejection and flux, resistance of the polarized layer and the resistance offered by the material deposited on the surface are also to be considered. This is because they effect flux and consequently may reduce the permeability of the membrane. From table 5.5 it can be noted that 15000 membrane provides lowest specific resistance and reasonably low polarized layer resistance at 680 kPa(100 psi) and 10 brix. However, at the same pressure and high feed concentration (16 brix); it is the 10000 which can be considered as the favourable membrane. But it should also be noted that the rejection of sucrose is comparatively higher(17.2%) using 10000 membrane

which is not favourable in terms of recovery of sucrose, whereas 20000 membrane can be advantageous in such conditions. From the above discussion one can, therefore, draw the following inferences :

1. A pressure of 680 kPa (100 psi) and feed concentration of 10 brix seem to be the favourable conditions as they gave high flux and reasonably low values of rejection of sucrose.
2. The 15000 membrane appears to show better results as compared to other two, in terms of high flux, low resistance and low rejection (at 680 kPa and 10 brix).
3. However, if the limed juice is to be treated at high concentration, then 20000 membrane appears to be favourable as it gives a reasonably high flux and does not comparatively offer much resistance.

It was thought to analyse the scheme of UF for the recovery of sucrose from limed cane juice with the help of above stated results. It is obvious that the choice for better scheme would depend upon high flux, low rejection of sucrose and low resistances against flow. However, a direct comparisons of all the schemes with the results were not possible as the results differed in units and particularly because of the choice of the membranes that varied in characteristics.

# **Chapter 6**

## **CONCLUSIONS**

Based on the experimental work carried out on the treatment of sugarcane juice using UF and also on the theoretical interpretation of the results, the following conclusions are drawn :

1. Liming of raw sugarcane juice with milk of lime to a pH of 8.0, at room temperature , followed by prefiltration removed approximately 20% of the total solids from the juice .
2. From the experimental results it was found that at stirrer speed of 600 rpm , 680 kPa (pressure) and 10 brix( feed concentration) are the most favourable operating conditions as they provided reasonably high flux and low rejection of sucrose in all cases.
3. From the comparative study of the three membranes viz. 10000, 15000 and 20000 MWCO membrane , the 15000 MWCO membrane was found most suitable. It not only provided high flux and low sucrose rejection at 680 kPa pressure and 10 brix feed concentration but offered low specific and polarized layer resistances.
4. In case, if the limed juice is to be ultrafilterd at high concentration (higher than 13 brix ) then the 20000 MWCO membrane would <sup>be</sup> more suitable. This <sup>is</sup> because at high concentration and high pressure it offers comparatively low resistances besides giving low sucrose rejection .

## **Chapter 7**

### **RECOMMENDATIONS**

The work is the first experimental study that is carried out in our lab for treatment of natural sugarcane juice, to recover sucrose using membrane separation processes (particularly UF). There were many limitations to the work and more detailed study was required to be undertaken. Hence, several recommendations and suggestions can be made for the development of a technique by which cane juice can be ultrafiltered in such a way as to give clear and transparent permeate of same sucrose content. Some of them are listed below :

1. The UF of limed juice was studied for small range of pressure and a fixed stirrer speed. Hence, it should be studied at different stirrer speeds and wide range of pressures.
2. A detailed study of flux decline behaviour and sucrose rejection of other sharp cut-off as well as diffuse cut-off membranes should be done in order to find a suitable membrane .
3. The present study was focused only on the recovery of sucrose however, the task of concentration sucrose by Nanofiltration (NF) and Reverse Osmosis (RO) should also be undertaken.
4. In the present work, it was observed that there was a deposition on the membrane surface that was detrimental to the flux as well as life of the membrane. To overcome this drawback, some experiments using cross-flow cell and vortex-flow cell should be conducted.
5. A theoretical model should also be developed to predict the flux, rejection and resistance apriori.
6. To reduce the load of highmolecular weight substances on membranes, some pretreatment methods should also be developed.

32. C. Bhattacharya and P. K. Bhattacharya, Flux decline analysis in UF of kraft black liquor, *J. Membrane Sci.*, **82** (1993) 1 - 14.
33. Jennifer M. Dias, Analysis of polarize layer resistance through secondary gel layer type in UF, *M.Tech. Thesis, IIT Kanpur*, (1993).
34. P. M. Bungery, H. K. Lonsdale and M. N. de Pinho, Synthetic membranes: science, engineering and application, *D. Riedel pub. Company, Holland*.
36. G. Belfort and F. W. Altena, Towards an inductive understanding of membrane fouling, *Desalination*, **47** (1983) 105.
37. J.G. Wijman, S. Nakao and C. A. Smolders, Flux limitation in UF : osmotic pressure model and gel layer model, *J. Membrane Sci.*, **20** (1984) 115.
38. J. G. Wijman, S. Nakao, T. W. A. NanDerberg, F.R. Troelster and C. A. Smolders, Hydrodynamic Resistance of concentration polarization layers in UF, *J. Membrane Sci.*, **22** (1) (1985) 117.
39. S. Nakao, J. G. Wijman and C. A. Smolders, Resistance to the permeate flux in unstirred UF of dissolved macromolecules, *J. Membrane Sci.*, **26** (1986) 165.
40. S. Nakao and S. Kimura, Analysis of solute rejection in UF, *J. Membrane Sci.*, **14** (1981) 32.
41. H. Niemi, T. RAimoaho and S. Palosaari, Modelling and simulation of UF and RO processes, *ACTA Polytechnica Scandinavica, Chem. Tech. and Met. Series*, No. 174, Helsinki (1986).
42. D. bhattacharya and M. R. Madadi, Separation of phenolic compounds by low pressure composite membranes: Mathematical model and numerical results, *AI Ch.E Symp*, **84** (261) (1988), 139.
43. J. M. Dicksono, The effect of solute membrane affinity on cyclic hydrocarbon water transport in pressure driver membrane separation processes, *J. Membrane Sci.*, **21** (1) (1984) 21.
44. D. D. Do and A. A. Elhassadi, A theory of limiting flux in stirred batch cell, *J. Membrane Sci.*, **2** (1)(1984) 114.
45. D. Bhattacharya, S. L.Kermode and M. C. Roco, Prediction of concentration polarization and flux behavior in RO by numerical analysis, *J. Membrane Sci.*, **48** (1990) 231.
46. Y. kao and Z. Yan, Dynamic modelling and simulation by single membrane parameters, *Chem. Eng. Comm.*, **59** (1987) 343.

47. V. Gekas and B. Hallstrom, Mass transfer in the membrane concentration polarization layer under turbulent cross flow critical literature review and adaptation of existing sherwood correlation to membrane operations, *J. Membrane Sci.*, **30** (1987) 153.
48. C. Belfort and N. Nagata, Fluid mechanics and cross flow filtration: some thoughts, *Desalination*, **53** (1985) 57.
49. M.A. Mazid, Separation and fractionation of macromolecular solution by ultrafiltration, *Sep. Sci. Technol.*, **23** (1988) 219.
50. G. B. Vander Berg and C. A. Smolden, Flux decline in UF processes, *Desalination*, **77** (1990) 101.
51. P. Punidas, M. Decloux and G. Trystran, Cross flow MF on ceramic inorganic membranes, *Industries Alimentaires et Agricoles*, **107** (1990) 615 - 623.
52. G. Jonson, Overview of theories for water and solute transport in UF/RO membranes, *Desalination*, **35** (1980) 21.

## **APPENDIX A**

### **A-1 Physical properties of limed cane juice**

All the properties of the limed juice were measured at 23°C.

#### **A.1.1. Determination of density - refractive index correlation**

From density- concentration data given in table A-1, following correlation was developed:

$$\rho = 9.87 \times 10^{-1} + 3.4908 \times 10^{-3} C + 1.00387 \times 10^{-4} C^2$$

where, C is concentration of the solution in RI(brix) and  $\rho$  density is in g/cc.

#### **A.1.2 Determination of viscosity - refractive index correlation**

From the viscosity-refractive index data given in table A-2, following correlation was developed:

$$\mu = 8.08896 \times 10^{-1} + 8.27988 \times 10^{-2} C + 6.72107 \times 10^{-3} C^2 + 2.85867 \times 10^{-4} C^3$$

where, C is cocentration in RI and  $\mu$  is viscosity in cP.

#### **A.1.3 Determination of concentration - refractive index**

Refractive index of limed cane juice at various concentration was measured at 23°C. The figure 5.1 shows the calibration curve of concentration versus RI of limed as well as raw cane juice. Table A-3 and table A-4 show the concentration - RI data for limed and raw juice.

RI (brix)	Density (g/cc)
1.6	0.990
3.0	1.00
5.0	1.005
6.8	1.021
9.2	1.022
11.2	1.034
13.6	1.053
16.3	1.071

**Table A.1**Refractive index - Density data for limed cane juice

RI (brix)	Viscosity (cp)
1.6	0.9274
5.0	1.084
6.8	1.148
9.2	1.246
11.2	1.278
13.0	1.379
16.0	1.612

**Table A.2**Refractive index - Viscosity data for limed cane juice

RI (brix)	Concentration (g/cc)
1.6	0.01162
3.0	0.02189
5.0	0.03642
6.8	0.04790
9.2	0.06120
11.2	0.08032
13.0	0.0782
16.5	0.1138

**Table A.3**Refractive index-Concentration data for limed cane juice

RI (brix)	Concentration (g/cc)
1.6	0.01
3.00	0.03
6.0	0.06
8.0	0.07
11.4	0.09
15.4	0.13
19.0	0.17

**Table A.4**Refractive index-Concentration data for raw cane juice

## APPENDIX B

To prove that refractive index (RI) follows additive law ,following experiment was conducted.

### Experimental procedure

Three different solutes - dextrose, sucrose and bovine serum albumin (BSA), were taken. They were separately dissolved in distilled water and the RI of the solutions were measured. Then two of the solutions were mixed and the RI was measured. After that all three solutions were mixed and RI was measured. Here it is to be noted that the amount of solutes taken were approximately in the same proportion as they are present in raw sugarcane juice. The results of the experiment are tabulated in table B.1. From the table one can observe that RI is an additive extensive property.

Dextrose RI (brix)	BSA RI (brix)	Sucrose RI (brix)	(Dextrose+Sucrose+BSA) RI (brix)
1.1	0.6	0.6	17.5
1.0	0.5	13.9	15.4
0.9	0.4	12.2	13.7
0.8	0.3	10.6	11.7
0.6	0.3	8.9	9.8
0.5	0.2	7.0	7.8

Table B.1Refractive indices of various solutes and their solutions

## APPENDIX C

A block diagram of the algorithm used to calculate the flux and polarised layer resistance at different time intervals during the experiment is given in figure C.1. The algorithm uses the osmotic pressure model to find the flux and resistance. The correlation used to find the change in osmotic pressure with change in the concentration at the membrane surface is given in equation given below. It can be observed that the correlation is only for sucrose. This is used because it is assumed that all the nonpermeable species that are completely rejected by the membranes offer no osmotic pressure as they are macromolecular substances. Secondly, out of all the low molecular weight the content of sucros is highest, hence osmotic pressure calculations are based on sucrose only.

$$\pi(c) = -\frac{RT}{V_w} \left[ \frac{\frac{100-c}{\mu_w} - \frac{4c}{\mu_s}}{\frac{100-c}{\mu_w} - \frac{3c}{\mu_s}} \right]$$

where,  $V_w$ = Partial molal volume of water ( $18.016 \times 10^{-6} m^3/mol$ )

$M_w$ =Molecular weight of water( $18.016 \times 10^{-3} kg/mol$ )

$M_s$ =Molecular weight of sucrose( $342.30 \times 10^{-3} Kg/mol$ )

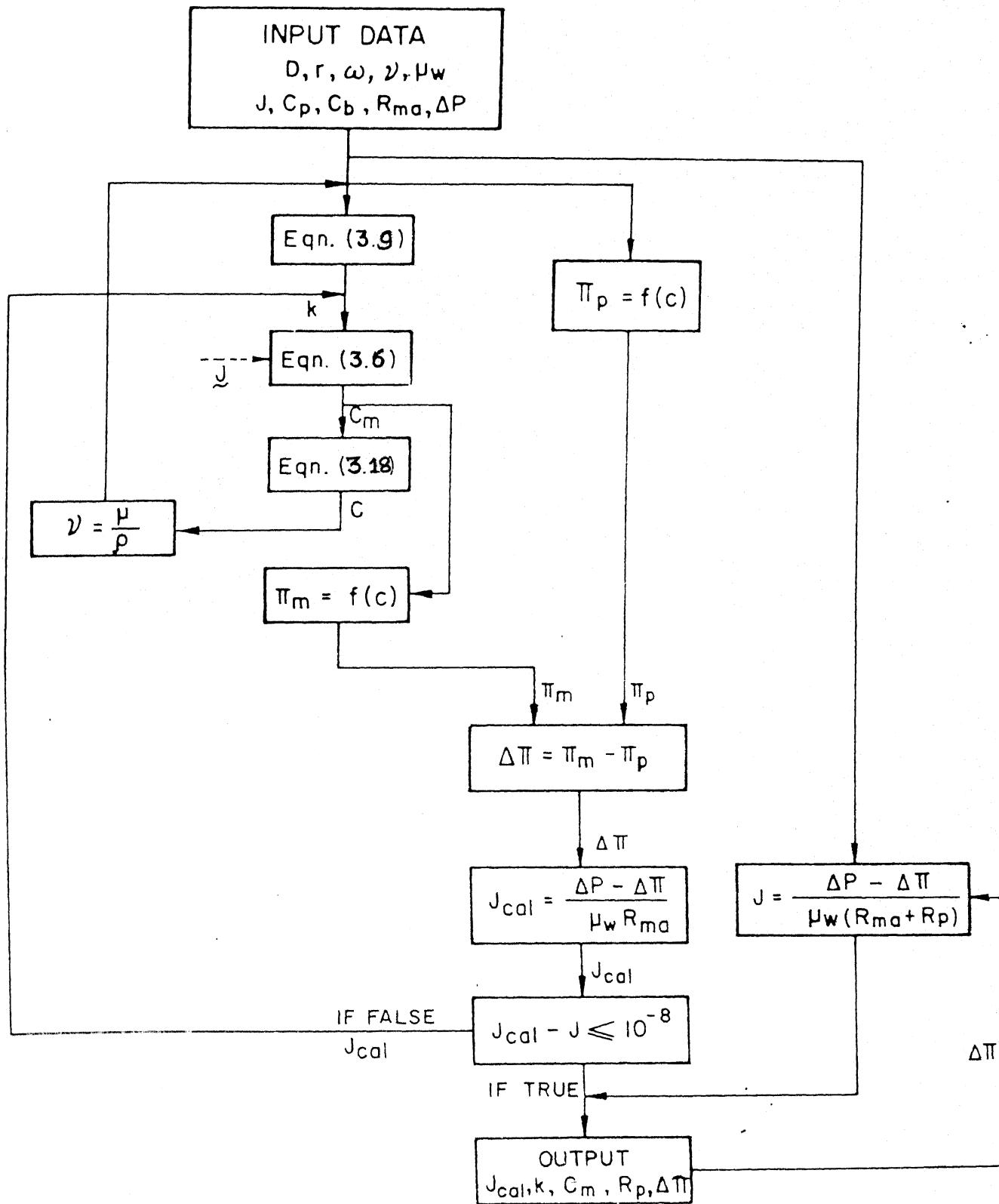


Fig. C.1 A block diagram for the algorithm.

## MEMBRANE 20000

RI(F) = 10 brix

$P = 476.19 \text{ kPa}$	$Rm = 7.968e13 /m$	$RI(P) = 8.6$	$P = 680.272 \text{ kPa}$	$Rm = 8.01e13 /m$	$RI(P) = 8.4$	$P = 884.353 \text{ kPa}$	$Rm = 7.45e13 /m$	$RI(P) = 8.0$
$t(s)$	flux(m/s)		$t(s)$	flux(m/s)		$t(s)$	Flux(m/s)	
$(x10e-2)$	$(x10e6)$		$(x10e-2)$	$(x10e6)$		$(x10e-2)$	$(x10e6)$	
0.0	5.98		0.0	6.93		0.0	7.95	
2.78	4.96		3.42	6.11		6.73	7.01	
6.00	4.68		7.20	5.21		10.67	6.57	
7.61	4.68		12.52	5.20		12.71	6.39	
10.86	4.59		16.01	4.78		14.86	6.01	
12.54	4.49		19.16	4.74		17.25	5.49	
14.24	4.49		22.34	4.74		22.05	5.35	
15.88	4.47		28.95	4.68		27.05	5.30	
17.88	4.42		22.45	4.55		29.45	4.75	
21.10	4.41							

RI(F) = 13 brix

$P = 476.19 \text{ kPa}$	$Rm = 8.02e13 /m$	$RI(P) = 11.8$	$P = 680.272 \text{ kPa}$	$Rm = 7.12e13 /m$	$RI(P) = 10.8$	$P = 884.353 \text{ kPa}$	$Rm = 7.62e13 /m$	$RI(P) = 10.0$
$t(s)$	Flux(m/s)		$t(s)$	Flux(m/s)		$t(s)$	Flux(m/s)	
$(x10e-2)$	$(x10e6)$		$(x10e-2)$	$(x10e6)$		$(x10e-2)$	$(x10e6)$	
0.0	7.50		0.0	7.98		0.0	8.05	
1.05	7.18		4.27	6.93		3.73	7.68	
2.24	6.33		8.80	6.44		7.49	6.65	
5.10	5.82		11.46	5.66		9.54	6.35	
7.43	5.80		16.77	5.56		15.60	6.23	
10.15	5.54		19.70	5.34		17.80	5.95	
13.05	5.34		25.30	5.01		19.98	5.91	
15.95	4.79		28.27	4.97		24.33	5.52	
19.40	4.63		36.90	4.67		29.65	4.86	
21.70	4.32		42.90	4.45		31.97	4.84	
24.52	4.20					37.05	4.60	

RI(F) = 16 brix

$P = 476.19 \text{ kPa}$	$Rm = 7.85e13 /m$	$RI(P) = 13.6$	$P = 680.272 \text{ kPa}$	$Rm = 8.12e13 /m$	$RI(P) = 13.0$	$P = 884.353 \text{ kPa}$	$Rm = 7.11e13 /m$	$RI(P) = 12.2$
$t(s)$	Flux(m/s)		$t(s)$	Flux(m/s)		$t(s)$	Flux(m/s)	
$(x10e-2)$	$(x10e6)$		$(x10e-2)$	$(x10e6)$		$(x10e-2)$	$(x10e6)$	
4.52	5.35		3.72	6.0		2.21	6.10	
6.92	5.0		9.34	5.10		4.48	5.60	
9.34	4.2		15.34	5.02		6.79	5.50	
11.82	3.89		18.35	5.0		9.06	5.55	
16.86	3.87		28.05	4.69		14.06	5.05	
19.39	3.86		45.01	4.37		17.0	5.05	
21.94	3.82		55.20	4.43		19.51	5.01	
24.47	3.81		59.07	4.11		22.33	4.35	
29.50	3.66		62.63	3.89		35.95	4.03	

The tables below give the calculated values of flux( $j_{cal}$ ) and polarised layer resistance( $R_p$ ) for each experiment.

MEMBRANE = 10000

RI(Feed)=10 brix

P=476.19kPa

$j_{cal}=1.19 \times 10^{-5} \text{ m/s}$

P=680.27kPa

$j_{cal}=1.70 \times 10^{-5} \text{ m/s}$

P=884.353kPa

$j_{cal}=2.23 \times 10^{-5} \text{ m/s}$

t (s)	$R_p(t)/\text{m}$	t (s)	$R_p(t)/\text{m}$	t (s)	$R_p(t)/\text{m}$
4.32e2	3.86e13	4.5e2	6.82e13	2.13e2	7.30e13
6.99e2	4.82e13	7.07e2	8.05e13	4.29e2	8.72e13
15.86e2	4.88e13	18.64e2	8.14e13	17.0e2	1.01e14
18.57e2	5.19e13	21.55e2	8.23e13	26.4e2	1.01e14
23.85e2	5.50e13	27.59e2	8.31e13	28.91e2	1.03e14
28.60e2	5.50e13	30.60e2	8.52e13	33.90e2	1.04e14
31.37e2	6.11e13	36.70e2	8.52e13	36.40e2	1.07e14
34.14e2	6.11e13	38.30e2	8.52e13	41.48e2	1.07e14
	6.11e13				

RI(Feed)=13 brix

P=476.19kPa

$j_{cal}=1.20 \times 10^{-5} \text{ m/s}$

P=680.272kPa

$j_{cal}=1.72 \times 10^{-5} \text{ m/s}$

P=884.353kPa

$j_{cal}=2.21 \times 10^{-5} \text{ m/s}$

t (s)	$R_p(t)/\text{m}$	t (s)	$R_p(t)/\text{m}$	t (s)	$R_p(t)/\text{m}$
0.0 e2	4.12e13	4.53e2	6.16e13	2.93e2	7.40e13
2.24e2	4.35e13	6.73e2	7.42e13	6.41e2	7.28e13
6.43e2	5.82e13	10.67e2	7.67e13	9.27e2	8.68e13
13.05e	6.28e13	12.71e2	7.91e13	12.79e2	9.70e13
15.95e2	7.35e13	17.25e2	8.30e13	16.48e2	9.81e13
19.40e2	8.28e13	22.05e2	9.30e13	23.93e2	1.04e14
24.52e2	8.64e13	24.44e2	9.62e13	31.43e2	1.05e14
27.51e2	8.70e13			35.30e2	1.08e14
34.69e2	8.70e13			39.14e2	1.08e14
38.28e2	8.70e13				

RI(feed)=16 brix

P=476.19kPa

$j_{cal}=2.21 \times 10^{-5} \text{ m/s}$

P=680.272kPa

$j_{cal}=1.71 \times 10^{-5} \text{ m/s}$

P=884.353kPa

$j_{cal}=1.27 \times 10^{-5} \text{ m/s}$

t (s)	$R_p(t)/\text{m}$	t (s)	$R_p(t)/\text{m}$	t (s)	$R_p(t)/\text{m}$
5.80e2	1.89e14	5.35e2	7.04e13	6.37e2	6.83e13
7.75e2	1.90e14	8.53e2	9.16e13	8.61e2	7.34e13
10.10e2	1.91e14	11.56e2	9.37e13	10.96e2	7.87e13
13.64e2	1.96e14	14.65e2	9.75e13	13.40e2	7.70e13
15.65e2	2.03e14	24.48e2	9.97e13	15.93e2	7.82e13
28.20e2	2.08e14	31.20e2	1.09e14	18.43e2	8.42e13
33.06e2	2.25e14	34.54e2	1.11e14	20.95e2	8.94e13
37.45e2	2.30e14	37.88e2	1.11e14	23.57e2	9.69e13
38.76e2	2.30e14				

MEMBRANE=15000

RI(F)=10 brix

P=476.19kPa

P=680.272kPa

P=884.353kPa

jcal=1.63e-5m/s

jcal=2.17e-5m/s

jcal=2.34e-5m/s

t(s)	Rp(t)/m
5.19e2	5.38e13
11.66e2	6.50e13
14.32e2	6.59e13
17.02e2	6.63e13
19.70e2	6.85e13
22.50e2	7.20e13
25.52e2	7.29e13

t(s)	Rp(t)/m
5.12e2	7.05e13
9.53e2	7.84e13
20.42e2	8.70e13
22.14e2	9.02e13
27.16e2	9.06e13
32.02e2	9.24e13

t(s)	Rp(t)/m
5.53e2	7.34e13
11.66e2	8.68e13
15.99e2	8.73e13
18.33e2	8.94e13
22.66e2	9.34e13
27.16e2	9.44e13

RI(F)=13 brix

P=476.19kPa

jcal=1.47e-5m/s

P=680.27kPa

jcal=1.41e-5m/s

P=884.353kPa

jcal=2.32e-5m/s

t(s)	Rp(t)/m
7.20e2	8.83e13
12.52e2	9.07e13
19.16e2	1.21e14
25.49e2	1.34e14
32.45e2	1.43e14
39.06e2	1.62e14
43.21e2	1.79e14
48.06e2	1.81e14

t(s)	Rp(t)/m
4.95e2	7.56e13
7.60e2	9.13e13
10.22e2	9.76e13
12.83e2	1.13e14
15.56e2	1.17e14
18.20e2	1.31e14
20.92e2	1.38e14
23.50e2	1.49e14

t(s)	Rp(t)/m
8.56e2	8.47e13
12.37e2	8.82e13
15.80e2	9.19e13
19.05e2	1.09e14
24.72e2	1.15e14
28.21e2	1.17e14
33.22e2	1.17e14

RI(F)=16 brix

P=476.19kPa

jcal=1.12e-5m/s

P=680.27kPa

jcal=2.17e-5m/s

P=884.353kPa

jcal=2.27e-5m/s

t(s)	Rp(t)/m
3.16e2	1.58e14
11.63e2	2.25e14
16.08e2	2.00e14
20.29e2	2.90e14
24.91e2	2.98e14
30.93e2	3.00e14
35.39e2	3.29e14
39.79e2	3.51e14
42.40e2	2.30e14
45.00e2	3.10e14

t(s)	Rp(t)/m
9.67e2	1.23e14
11.9e2	1.41e14
13.99e2	1.60e14
17.48e2	1.68e14
22.83e2	1.74e14
28.98e2	1.75e14
34.20e2	1.82e14
39.54e2	1.95e14
42.18e2	2.01e14

t(s)	Rp(t)/m
3.00e2	1.17e14
6.21e2	1.52e14
13.18e2	1.53e14
16.41e2	1.51e14
19.71e2	1.55e14
23.08e2	1.58e14
26.45e2	1.59e14
33.28e2	1.59e14
40.25e2	1.59e14

## MEMBRANE=20000

RI(F)=10 brix

P=476.19kPa

jcal=5.97e-6m/s

P=680.272kPa  
jcal=8.49e-6m/sP=884.35kPa  
jcal=1.19e-5m/s

t(s)	Rp(t)/m
2.78e2	2.20e13
6.00e2	2.20e13
7.61e2	2.39e13
10.86e2	2.63e13
12.54e2	2.63e13
14.24e2	2.67e13
15.88e2	2.79e13
17.88e2	2.82e13
21.10e2	2.82e13

t(s)	Rp(t)/m
3.42e2	3.14e13
7.20e2	5.05e13
12.52e2	5.07e13
16.01e2	6.16e13
19.16e2	6.34e13
22.34e2	6.53e13

t(s)	Rp(t)/m
6.73e2	5.17e13
10.67e2	6.01e13
12.71e2	6.39e13
14.86e2	7.26e13
17.25e2	8.66e13
22.05e2	9.08e13
27.05e2	9.24e13

RI(F)=13 brix

P=476.19kPa

jcal=5.94e-6m/s

P=680.27kPa  
jcal=9.55e-6m/sP=884.35kPa  
jcal=1.16e-5m/s

t(s)	Rp(t)/m
1.05e2	1.62e12
2.24e2	1.90e12
5.10e2	5.75e12
7.43e2	8.97e12
10.15e2	1.92e13
13.05e2	2.26e13
15.95e2	3.00e13

t(s)	Rp(t)/m
4.27e2	2.70e13
8.80e2	3.44e13
11.46e2	4.90e13
16.77e2	5.12e13
19.70e2	5.63e13
25.30e2	6.46e13
28.27e2	6.57e13
36.90e2	7.45e13

t(s)	Rp(t)/m
3.73e2	3.90e13
7.49e2	5.68e13
9.54e2	6.31e13
15.60e2	6.58e13
17.80e2	7.24e13
19.98e2	8.40e13
24.33e2	1.06e14
29.65e2	1.07e14

RI(F)=16 brix

P=476.19kPa

jcal=6.06e-6m/s

P=680.27kPa  
jcal=8.38e-6m/sP=884.353kPa  
jcal=7.25e-6m/s

t(s)	Rp(t)/m
4.52e2	1.05e13
6.92e2	1.67e13
9.34e2	3.49e13
11.82e2	4.39e13
16.86e2	4.45e13
19.39e2	4.49e13
21.94e2	4.62e13
24.47e2	4.65e13

t(s)	Rp(t)/m
3.72e2	3.22e13
9.34e2	5.22e13
15.34e2	5.43e13
18.35e2	5.49e13
28.05e2	6.38e13
45.01e2	7.45e13
55.20e2	7.24e13
59.07e2	8.43e13

t(s)	Rp(t)/m
2.21e2	2.24e13
4.48e2	2.56e13
6.79e2	3.59e13
9.06e2	3.88e13
14.06e2	3.73e13
17.00e2	5.31e13
19.51e2	5.45e13
22.33e2	8.13e13

MEMBRANE 15000

RI(F) = 10 brix

$P = 476.190 \text{ kPa}$	$P = 680.272 \text{ kPa}$	$P = 884.353 \text{ kPa}$	
$Rm = 2.930e13 /m$	$Rm = 3.13e13 /m$	$Rm = 3.781e13 /m$	
$RI(P) = 8.2$	$RI(P) = 8.0$	$RI(P) = 7.8$	
$t(s)$ ( $\times 10e-2$ )	$Flux(m/s)$ ( $\times 10e6$ )	$t(s)$ ( $\times 10e-2$ )	
2.63	5.73	2.36	6.68
5.19	5.05	5.12	6.20
11.66	5.00	9.53	5.75
14.32	4.98	20.42	5.60
17.02	4.98	22.14	5.58
19.70	4.87	27.16	5.50
22.50	4.70	32.02	5.47
25.52	4.66		
28.55	4.60		

RI(F) = 13 brix

$P = 476.19 \text{ kPa}$	$P = 680.272 \text{ kPa}$	$P = 884.353 \text{ kPa}$	
$Rm = 4.068e13 /m$	$Rm = 4.81e13 /m$	$Rm = 3.812e13 /m$	
$RI(P) = 11.4$	$RI(P) = 10.0$	$RI(P) = 9.6$	
$t(s)$ ( $\times 10e-2$ )	$flux(m/s)$ ( $\times 10e6$ )	$t(s)$ ( $\times 10e-2$ )	
0.0	3.95	0.0	5.63
7.20	3.87	4.95	5.50
12.52	3.10	7.60	4.88
19.16	2.87	10.22	4.67
25.49	2.71	12.83	4.21
32.45	2.45	15.56	4.11
39.06	2.25	18.20	3.87
43.21	2.23	20.92	3.66
		23.50	3.45
		26.02	3.39

RI(F) = 16 brix

$P = 470.19 \text{ kPa}$	$P = 680.272 \text{ kPa}$	$P = 884.353 \text{ kPa}$	
$Rm = 4.256e13 /m$	$Rm = 6.39e13 /m$	$Rm = 7.784e13 /m$	
$RI(P) = 12.8$	$RI(P) = 12.0$	$RI(P) = 11.6$	
$t(s)$ ( $\times 10e-2$ )	$Flux(m/s)$ ( $\times 10e6$ )	$t(s)$ ( $\times 10e-2$ )	
0.0	2.26	4.41	4.40
3.16	2.38	9.67	3.95
11.63	1.78	11.92	3.56
16.08	1.66	13.99	3.41
20.29	1.43	17.48	3.31
24.91	1.40	22.83	3.29
30.93	1.39	28.98	3.19
35.39	1.28	34.20	3.0
39.79	1.21	39.54	2.93
42.40	1.95		
45.0	1.35		
47.80	1.08		

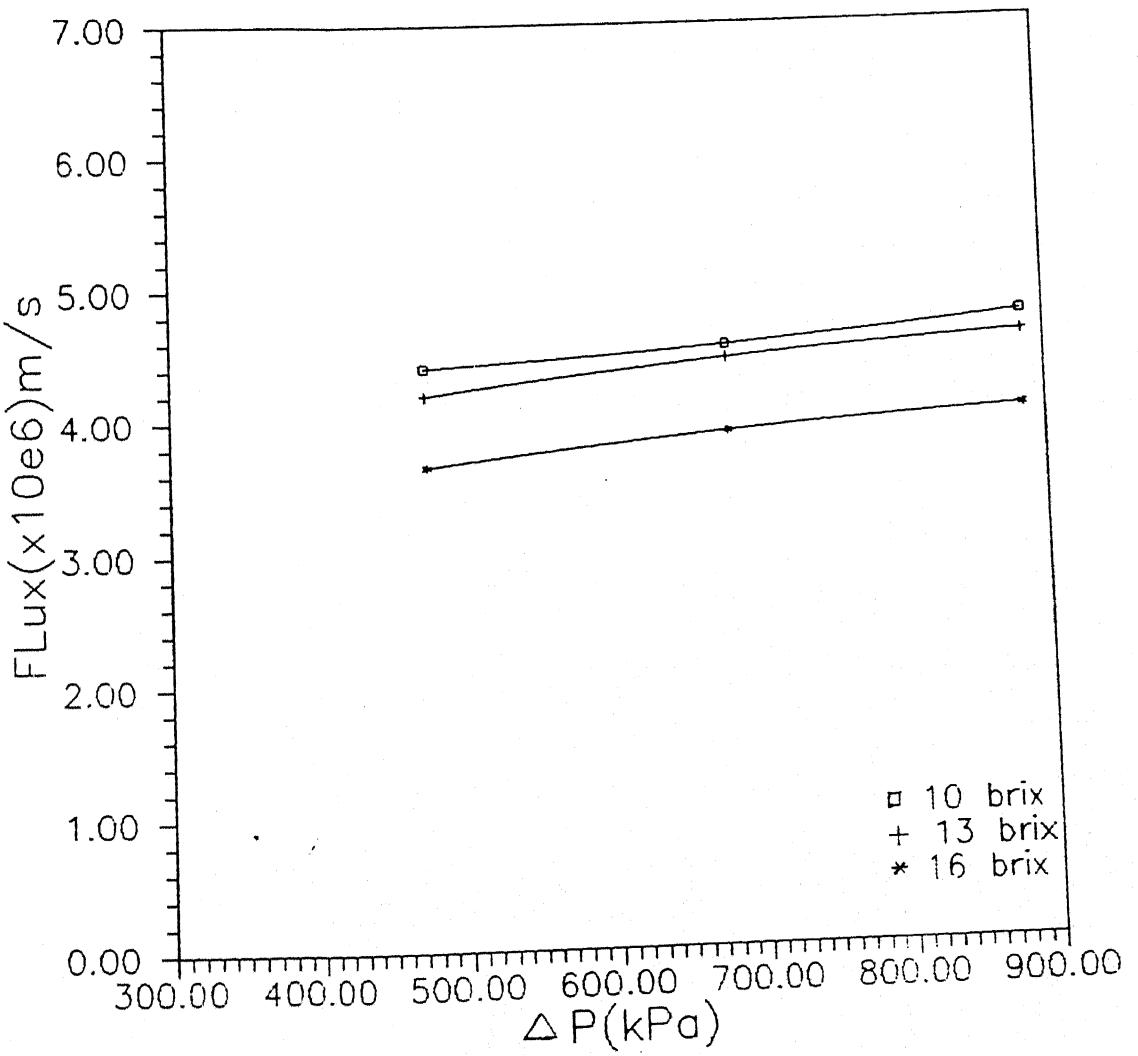


Fig 5.4 Effect of pressure on permeate flux for a 20000 membrane

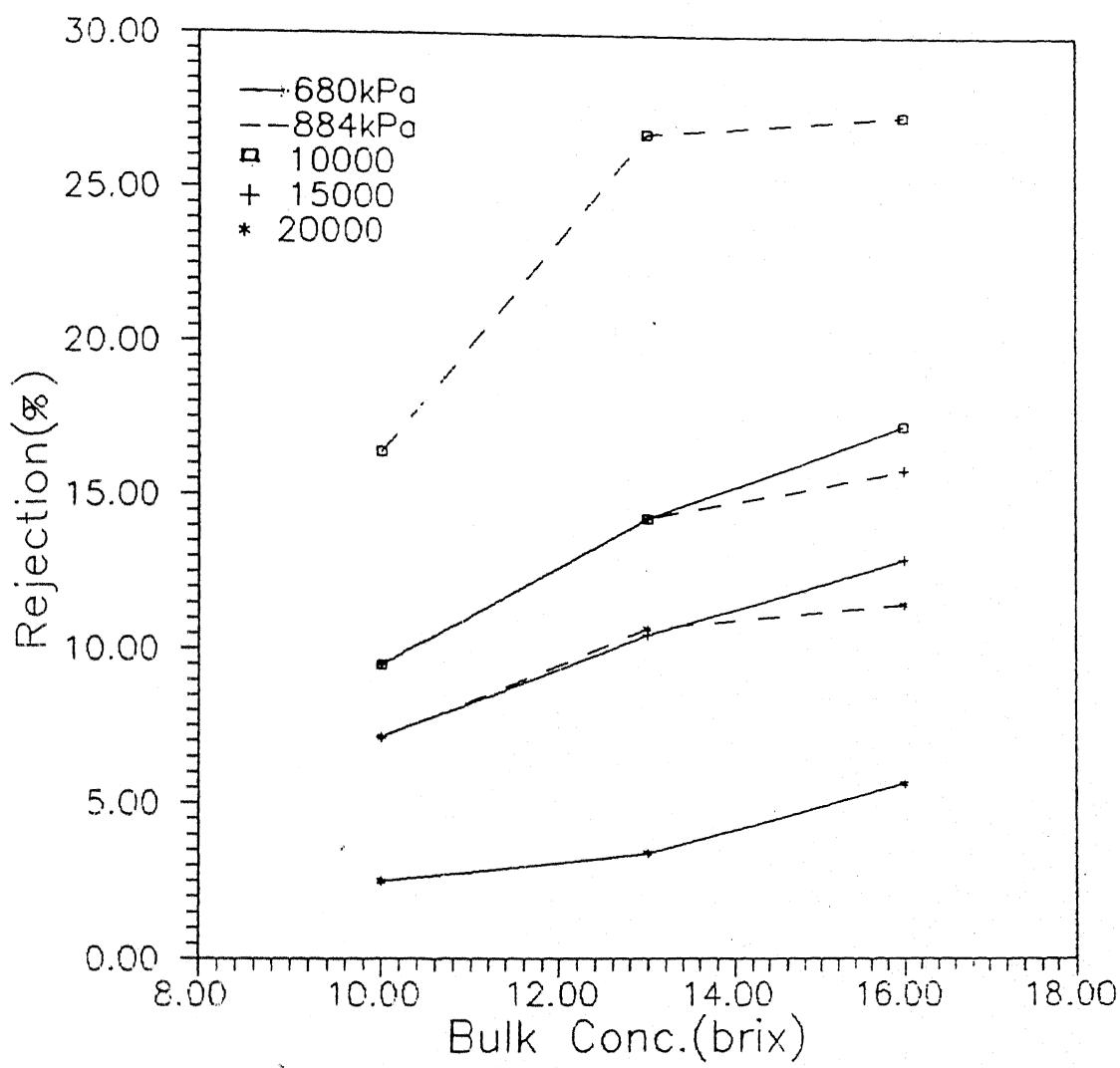


Fig 5.12 Effect of bulk concentration on sucrose rejection

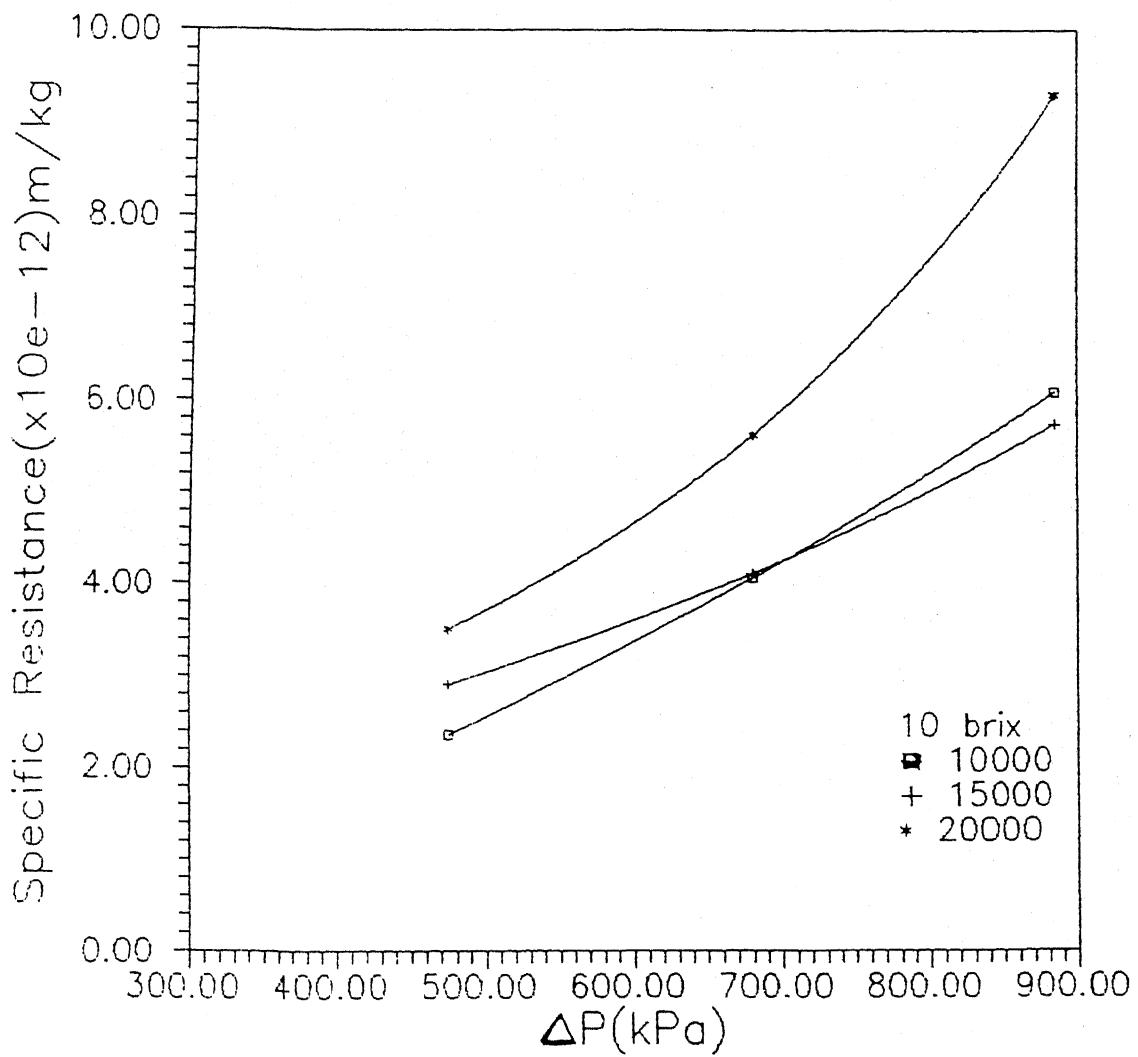


Fig 5.19 Effect of pressure on specific resistance

16. Arunabho Sahu, Modelling of ED process for impurity removal ( $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ ) from sugarcane juice after liming, *M.Tech Thesis, IIT Kanpur*.
17. H. Meade and G. Spencer, Cane sugar hand book, Wiley, NY, (1963).
18. R. L. Goldsmith, Macromolecular ultrafiltration with micro porous membranes, *Ind. Eng. Chem. Fundamental*, **10** (1971)113.
19. S. Shridhar and P. K. Bhattacharya, Limiting flux phenomenon in ultrafiltration of black liquor, *J. Membrane Sci.*, **57** ( 1991)187 – 208.
20. T. K. Poddar, R. P. Singh and P. K. Bhattacharya, Ultrafiltration flux and rejection characteristics of black liquor and polyethylene glycol , *Chem. Eng. Comm.*, **75** (1989) 39 - 45.
21. S. Dasgupta and P. K. Bhattacharya, Comparative limiting flux analysis of black liquor with polyethylene glycol in ultrafiltration, *Chem. Eng. Comm.* , **93** (1990) 193 - 210.
- 22.R. E. Treybal, Mass transfer operations , *Mcgraw Hill* , New York (1955).
- 23.M.R. Doshi, Limiting flux in the ultrafiltration of macromolecular solutions, *ACS Symposium Series*, no. **281** (1985) 209 - 215.
24. W. F. Blatt, A. Dand , A. S. Michael and L. Neilsen, Solute polarisation and cake formation in membrane ultrafiltration : Causes, consequences and control techniques, *Membrane Sci. Technol.*, J. E. flinn ed., Plenum, New York (1970) 47 - 73.
25. W. S. Winston Ho and K. K. Sirkar, Membrane Handbook , *Van Nostrand Reinold Publishing*, New York (1992).
- 26.D. B. Purchas, Industrial filtration of liquids, *Leonard Hill Books*, London, U K(1971).
27. M. E. Chudacek and A. G. Fane, The dynamics of Polarisation in unstirred and stirred ultrfiltration, *J. Membrane Sci.*, **21** (1984) 145- 160.
28. P. Aimar, C. Taddei, J. Lafaille and V. Sanchez, Mass transfer limitations during ultrafiltration of cheese whey with inorganic membranes, *J. Membrane Sci.* , **38** (1988) 203 - 221.
29. J. M. Coulson and J. F. Richardson, Chemical engineering, 7<sup>th</sup> edn., vol.2, *Pergaman press*, (1967).
30. S. Kimura and S. I. Nako, Fouling of cellular acetate tabular reverse osmosis modules treating industrial water in Tokyo, **17** (1975) 67.
31. C. Bhattacharya and P. K. Bhattacharya, Prediction of timing flux in UF of kraft black liquor, *J. Membrane Sci.*, **72** (1992) 137 - 147.